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THE BRITISH JOURNAL OF METALS

University of Michigan

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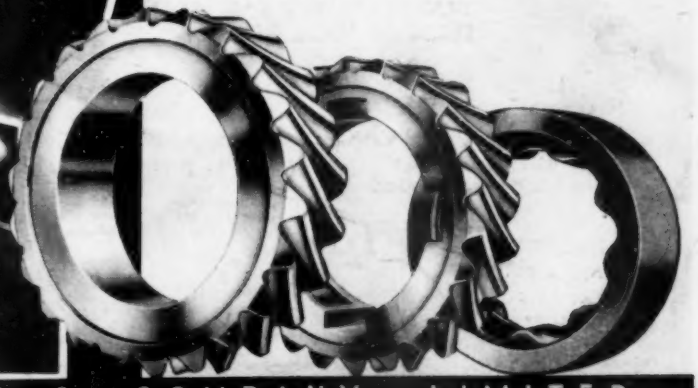

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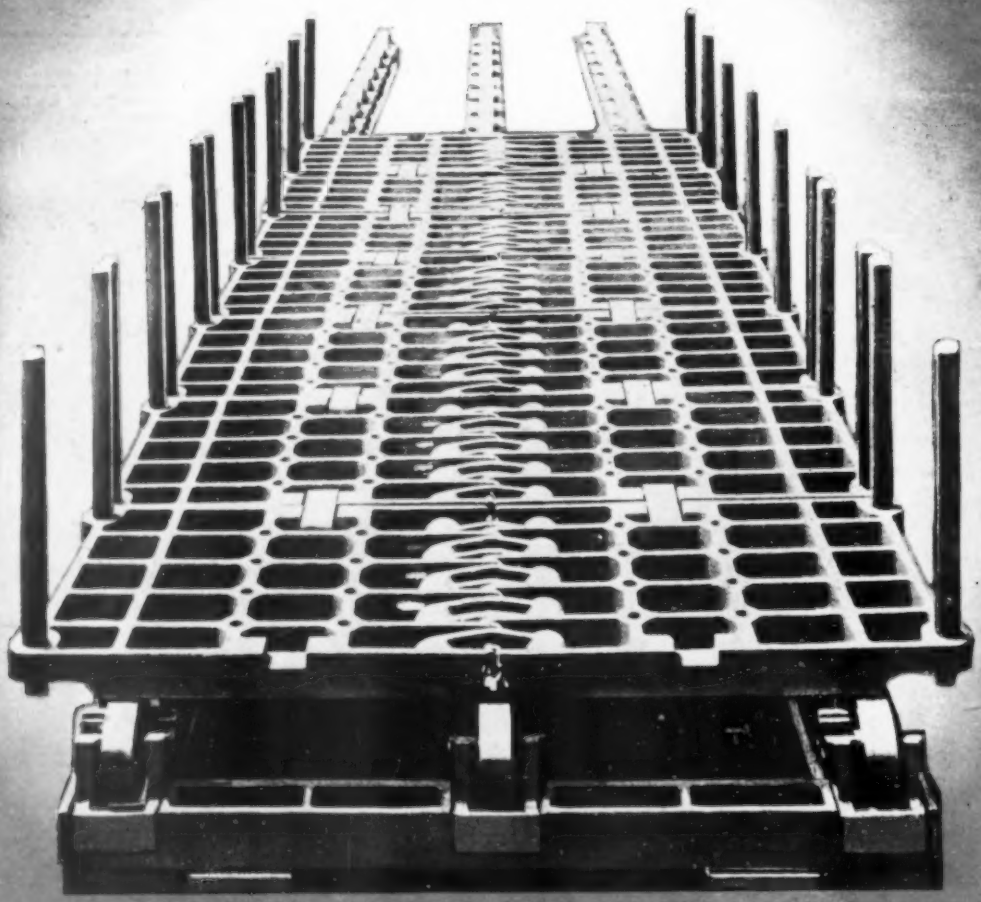

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The British Journal of Metals

(INCORPORATING THE METALLURGICAL ENGINEER)

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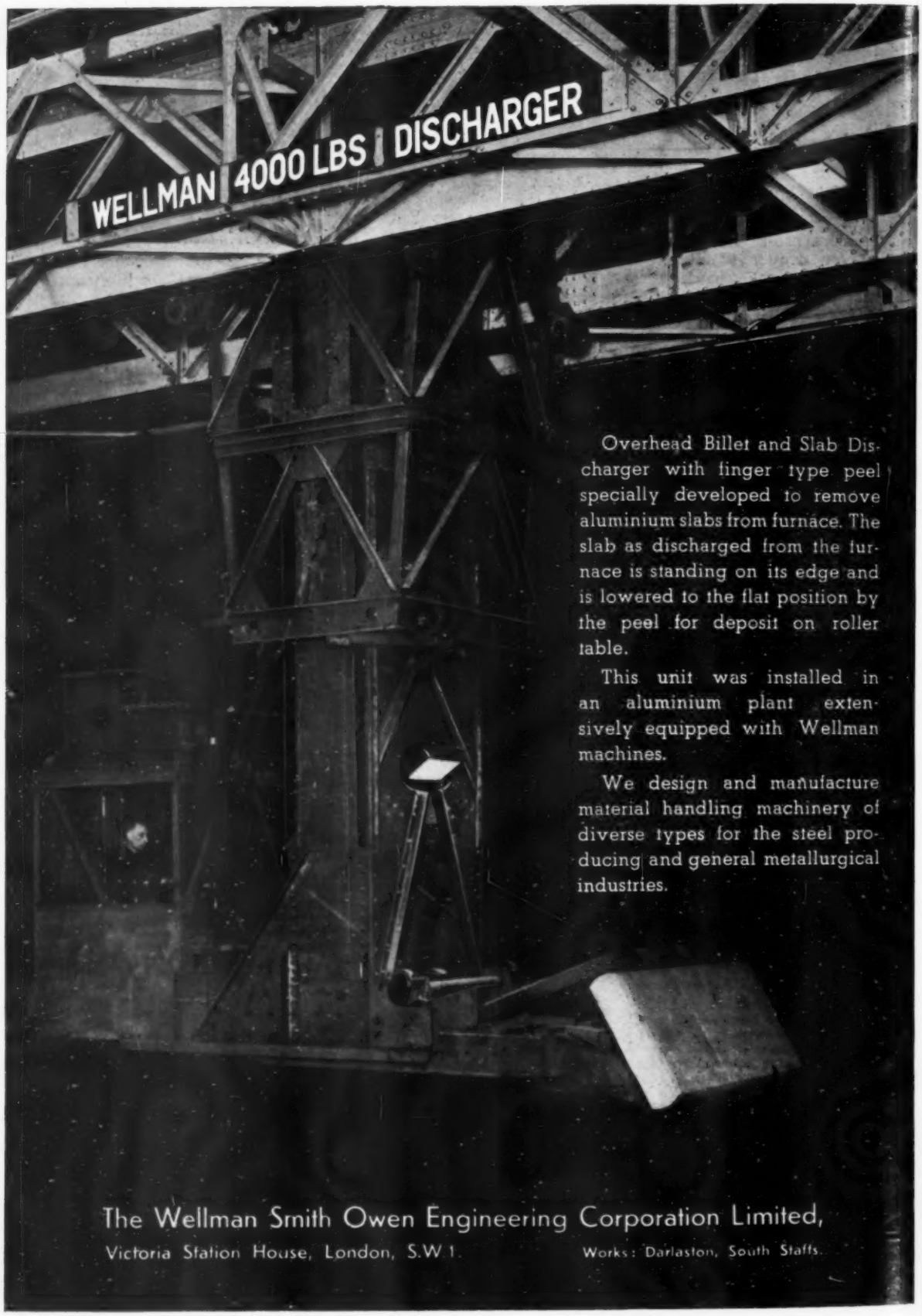

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METALLURGIA

THE BRITISH JOURNAL OF METALS.

INCORPORATING "THE METALLURGICAL ENGINEER."

MAY, 1947 VOL. XXXVI No. 211

Britain's Shop Window

THE British Industries Fair was born during the first great war, commencing in 1915, and held annually without interruption until 1939. During the second great upheaval it was suspended. But even in those dark days its revival was being considered. In 1943 a Committee was appointed to consider post-war export trade. It recommended that the British Industries Fair should be resumed as soon as possible, but in any case within two years of the end of the war.

When this view was accepted it was decided to call together the pre-war Advisory Committee. This Committee, which was elected by exhibitors, existed, before the war to advise the Department of Overseas Trade on all major and minor questions arising in the organisation and conduct of the Fair. It met in November, 1944, and after considering the foreseeable difficulties, recommended that the revival of the Fair should be planned for 1947. As the main emphasis of post-war Fairs would be on export, and as the potential contribution of many industries to export had changed during the war, it was recommended that a new Advisory Committee be formed of representatives from trade organisations. In this way it was considered that the new British Industries Fair could be planned to be widely representative of United Kingdom industry and thus make a maximum contribution to post-war export trade. Ultimately a new Committee of 64 members was appointed, under the Chairmanship of Mr. A. E. Welch, Under Secretary, Export Promotion Department, and in consultation with industry and the Government it was decided that May, 1947, would be the earliest suitable date.

That it has been possible to adhere to this decision, despite a fuel crisis which has necessitated emergency measures of great severity, is due to the remarkable support of industry for a Trade Fair after only 21 months of peace and there are numerous indications that this event will form the spear head of the national drive for increased export trade. But, after a lapse of eight years, this Fair has a much wider purpose than the immediate placing of orders. Although Britain's industrial recovery has been unsurpassed by that of any other nation, it is now well known that owing to difficulties in some industries, deliveries will be delayed. However, at the Fair, overseas buyers will be able to consider Britain's progress in relation to parallel difficulties throughout the world. They will be able to discuss these problems with the actual manufacturer. When the Fair was held in successive years the renewal of old friendships was a feature and, while the opportunity to meet old friends will continue to be a predominating feature, a lapse of eight years, during which a disastrous war was waged, will have effected many changes in the overseas buyers as well as the representatives of British manufacturers. Old friends will be keenly sought, however, and fresh contacts will be made while reviewing the many unique advances in industrial technique which have resulted from experience gained in war. In many cases a buyer will see innovations before they can be reproduced in quantity.

No previous British Industries Fair has offered the overseas buyer so many developments to see and discuss with the manufacturer. A record number of 3,116 manufacturers display their products. In the London section there are 1,272 exhibitors at Olympia and 866 at Earls Court. Exhibits at Olympia include: Chemicals; jewellery and electro-plate; watches and clocks; paper and stationery; scientific, optical and photographic goods; fancy goods; toys and games, etc. At Earls Court are textiles, the largest single section in the Fair, and among others leather, plastics, foodstuffs, etc. The products of the engineering and hardware trades are shown at Castle Bromwich, Birmingham, where there are 978 exhibitors. It is in connection with this latter section that a review is given elsewhere in this issue, where an effort has been made to direct attention to some important developments in the ferrous and non-ferrous metals fields and in plant and equipment used in various trades. It will be appreciated that, in some instances, important and interesting exhibits are omitted because adequate information is not available at the time of going to press.

Although this post-war Fair, the first to be held in this country, is primarily concerned with the development of Britain's export trade, the exhibits will prove to be a revelation to the home buyer as well as to the overseas buyer, indicating as it does the swiftness of Britain's industrial reconversion, the technical advances gained from wartime experience, and the vigour with which industry has applied itself to export demands. But the need for the acquisition of foreign exchange to enable Britain to maintain the flow of imports will prevent many products exhibited being available to home buyers until such time as a better balance between imports and exports can be attained. However, exports, as the purchasing medium for imports, are just as much part of the home consumers standard of living as are the goods Britain produces and sells in her home market. They provide the home consumer with some of his most essential needs, mainly food and raw materials, and although some apparent sacrifice will be involved, they are essential for the standard of life Britain aims at achieving.

This Fair is perhaps unique in two major aspects. It is entirely national in character, and only goods produced in the United Kingdom or the Commonwealth and Empire may be shown. In the second place, participation in it, with the single exception of the textile section, is confined to the manufacturers of the actual articles exhibited. It can be regarded as the world's greatest national trade fair at which Britain will show an urgent desire to fulfil the requirements of overseas markets.

£50,000 to Sheffield University

IT can now be announced that the Staveley Coal & Iron Co., Ltd. has promised to give to the University of Sheffield, by way of a seven-years' covenant, the sum of £50,000, to be applied towards the cost of the new department of chemistry. This is the largest gift yet received by the University from any firm, and it provides convincing evidence of the importance the directors of this Company attach to the advancement of academic knowledge as the essential basis of all really progressive industrial development.

Here we have an old Company, whose works have existed since the latter part of the 17th century, taking the farsighted view that the encouragement of free University research in all branches of scientific chemistry will in the long run benefit both the Company and industry as a whole. The directors realise that discovery and invention do not spring full grown from the brains of men, but are the practical results of long and arduous work. This work involves systematic exploration and many investigations, which avoid confusion, reduce the possibility of premature conclusions, indicate the often unexpected influence of one factor upon another and results in the final application of a discovery or development to the benefit of mankind. Obviously facilities must be provided to enable such work to proceed, and the provision of new chemistry buildings is one of the most urgent parts of the post-war plan of the University of Sheffield, towards the cost of which the Staveley Company is making this magnificent gift.

The Staveley Company has shown consistent progress and development throughout its long existence. Owing to the vision of the late Charles Markham, it was one of the pioneers in the manufacture of foundry iron and the large plant known as the Devonshire Works commenced operations in 1907. The Company installed its own coking installation, and an extensive recovery plant for by-products, and probably went further in this respect than any other Company in Britain as it constantly strove to be integrated and self-sufficing. The manufacture of sulphuric acid was commenced on an extensive scale to supply the Companys own needs and the surplus was marketed. It carries the process of tar distillation to its ultimate limit, and has extended into the field of dye intermediates and the production of nitro benzene and aniline oil. These and many other ancillary products are directly traceable to the Company's vertical and horizontal structure, all of which are founded on its control of the basic materials, coal and extensive iron-ore properties.

All these developments have tended to focus the Company's attention on the value of research as the basis of future potential development. Recently, the Staveley Company acquired the firm of Bradley & Foster, Ltd., which possesses enlightened research departments under the control of Dr. J. E. Hurst, under whose guidance an ambitious and wide programme of research activity is in course of development in both the metallurgical and chemical fields.

The Company envisage a far-reaching programme of extension in the manufacture of chemicals and, with this in view, not only has the building up of its own research department been decided upon but the diretors are convinced of the beneficial results likely to accrue from a closer collaboration with work carried on in Universities. Obviously, the University most closely related to the Company's activities is the University of Sheffield, and it is with this object that this handsome contribution has been made to the cost of the new building.

The new chemistry department will be close to the present University buildings in Western Bank, and has been designed for teaching and research in all the main branches of chemical science. Its eventual cost will exceed £400,000, and it will be large enough for all work in chemistry when the University attains its projected number of 3,000 full-time students in all faculties. In recognition of the Staveley Company's gift, the new building is to be known as "The Staveley Laboratories."

The Jubilee of the Electron

Prof. Thomson's Genius

FIFTY years ago, on April 30th, 1897, the British physicist, Prof. J. J. Thomson (later Sir Joseph Thomson) made, at an evening discourse at the Royal Institution in London the first public announcement of the existence of the electron, and the first approximate estimate of its mass. Cathode rays had been extensively studied in many laboratories for the previous fifteen or twenty years, and their peculiar properties had been widely discussed. It was Thomson's genius, which enabled him to unravel the confusing, and almost contradictory clues which the experiments afforded as to the nature of the phenomenon, to grasp what was important and what was secondary in the evidence, and to make the final experiment which demonstrated, beyond all reasonable doubt that the cathode rays consisted of negatively charged particles, all alike, and all much smaller than the smallest particle hitherto known to science, the atom of hydrogen.

It is entirely typical of the upward sweep of Thomson's genius, that in the same lecture, almost before the new infant electron had uttered its first cry, and certainly before it had been accepted into the family of scientific facts, he foreshadowed the electron theory, of matter, which has dominated physical research for the last fifty years. Modern physics, with all that it stands for, may be said to have originated with the unveiling of the electron, fifty years ago. To Thomson the world owes much: not merely for the original discovery, which probably could not have been long delayed, but still more for the brilliancy with which he led the researches of the succeeding twenty years, and the selfless devotion he gave to the training of the men who were to follow him.

The electron is now playing an active and indispensable part in almost every home and every industry. It runs our wireless sets, and plays an active part in television, radar, and many systems of industrial control. The electron industry is, we believe, already the seventh largest in the world, and its possibilities are only just now beginning to be developed.

The Institute of Physics and the Physical Society, in collaboration with the Institution of Electrical Engineers, is arranging to mark this Jubilee by a series of lectures and other functions in London on September 25th and 26th next: an exhibition demonstrating the great influence this discovery in pure physics has had on the life of the community will be opened at the Science Museum, London on September 26th and will remain open for about three months.

The Reduction of Arsenic Trioxide by Carbon and Carbon Monoxide

By R. C. Vickery, M.Sc., Ph.D. and R. W. Edwards

Results from an experimental investigation are described which show that yield of metallic arsenic, by the reduction of arsenic trioxide with carbon, does not apprcach that expected in theory. It is suggested that this is due to the difference in specific heats and vaporisation temperatures of the two components, and that the replacement of arsenic trioxide by arsenic compounds of lower vapour pressures may have some effect upon the yield.

THE preparation of elemental arsenic by sublimation from a mixture of arsenic trioxide and charcoal, rarely if ever, affords yields of more than 65% theoretical as g ven by the equation :—

$$2As_2O_3 + 3C \rightarrow 4As + 3CO_2 \quad (1)$$

More generally is approached a condition approximating :

$$2As_2O_3 + 3C \rightarrow 2As + As_2O_3 + CO_2 + CO + C \quad (2)$$

This second equation cannot claim stoichiochemical accuracy, but is based on observations in practice, the residue remaining after heating a charge to 800° C. consisting of carbon and unvolatilised arsenic trioxide with a little metallic arsenic—the sublimate shows variation in composition according to position in the condenser tube, from approximately 98% metallic arsenic with 2% As_2O_3 at the proximal end and approximately 50% As_2O_3 and 50% C at the distal end.

It was in order to investigate this incomplete reaction that the investigations here recorded were undertaken.

It may be initially considered that such an incomplete reaction as is indicated is only to be anticipated in view of the differences between the temperatures of sublimation of the As_2O_3 and oxidation of the carbon. The residual As_2O_3 could be considered to be the monoclinic allotrope of the trioxide, and hence to require a higher temperature for volatilisation. Although the first of these premises may be considered valid, at the temperature employed—*ca.*800° C.—it is difficult to believe that even the monoclinic phase of the trioxide with its sublimation temperature of 315° C. would remain unvolatilised. The composition of the sublimate at the distal end of the condenser is also indicative of incomplete reaction, as although a portion of the As_2O_3 may be considered to be due to oxidation of the arsenic vapour, it is obvious that the major portion represents sublimed, unattacked As_2O_3 which has a lower critical temperature than has elemental arsenic. An attempt to reduce this loss due to pre-reaction volatilisation was made by adding sodium carbonate to the trioxide-carbon mixture, it being assumed that the formation of sodium arsenate and arsenite in the reaction crucible would, due to the lower vapour pressures of these compounds as compared with As_2O_3, retard volatilisation of the trioxide until the carbon had reached a temperature high enough for quantitative reaction to take place. Some improvement in yield—*ca.*15%—was thus obtained, but the residue consisted of the sodium compounds of arsenic, with a lower total As content, whilst the percentage of arsenic in the proximal sublimate remained unchanged and that in the distal sublimate lowered.

Little information was found in the available literature which had any bearing on the subject in hand, and even the few widely disseminated papers found were only of general applicability, no specific references being observed.

The main points of importance are those relating to the temperatures of volatilisation of the allotropic forms of arsenic trioxide and of the allotropes of elemental arsenic. According to Mellor[1] the major forms of the trioxide have the following properties :—

Amorphous : S.G. 3·74 ; M.Pt., 200° C. ; Vpn. Pt., 275°–315° C., transforming in presence of moisture at 100° C. to the octahedral form.

Octahedral : S.G. 3·69 ; M.Pt., 251°–275° C. under own vapour pressure, but normally sublimes without fusion.

Monoclinic : S.G. 3·85 ; M.Pt., *ca.*315° C.

In addition to these allotropic modifications, Smit and Beljaars[2] have indicated the existence of three further phases ; their work was, however, based on vapour pressure determinations alone, and has yet to be confirmed. Generally, we had observed in contradiction to the above data, that presublimation fusion only occurred with the vitreous modification, whilst with the crystalline allotropes sublimation occurred before fusion.

The allotropic forms of elemental arsenic also show a fair degree of variation :

α—arsenic (yellow) : soluble in CS_2 ; unstable ; S.G. 3·7.

β—arsenic (black) : insoluble in CS_2 ; less stable than the γ form to which it transforms at 360° C. ; S.G.4·7.

γ—arsenic (grey) : insoluble in CS_2 ; stable, and ordinary form, volatilises slowly at 100° C. and rapidly at 450° C. ; S.G. 5·73.

Reduction of As_2O_3 in an atmosphere of CO is given by Mellor[1] as taking place at temperatures as low as 60° C., but combination of the carbon with oxygen (either from the trioxide or the residual air in the reaction crucible) can hardly be anticipated below 700° C.

The main points, therefore, which were considered to require investigation were :—

(i) Temperature of reaction between the allotropic forms of As_2O_3 and carbon.

(ii) Effect of inclusion in the charge, of substances lowering the vapour pressure of As_2O_3.

(iii) Employment of carbon monoxide gas in place of carbon for the reduction.

As is common with gas reactions or reactions resulting in the formation of gaseous products, many factors must influence the data obtained, but it was considered that by initial standardisation of the apparatus and procedures employed correlatable results could be obtained. The

1 *Compr. Treat. Inorg. & Theoret. Chem.*, Vol. IX.
2 *Proc. Acad. Sci.*, Amsterdam, **34**, 1,141, 1931.

materials employed also required standardisation—particularly the carbon employed as it has been well shown[3] that the source and mode of production of this material have great influence on the progress of reactions in which it takes part.

Apparatus and Materials

The advantages of the thermal conductivity method of gas analysis together with the success which has attended its applications led to a consideration of the adoption of this technique to the problems under investigation, as it was obvious that the reactions to be examined would exhibit changes in the composition of the gas phase present which would be thus measurable.

The principles of the thermal conductivity method of gas analysis will require no explication, having been fully dealt with by Barr[4], Hurst & Rideal[5] and many others. The application of the technique to this investigation may be indicated thus : as may be seen from the above equations, the non-condensable gaseous products of the reduction are CO, and eventually CO_2, the thermal conductivities (k) of air, CO and CO_2 are respectively $5{\cdot}68 \times 10^{-5}$, $4{\cdot}99 \times 10^{-5}$, and $3{\cdot}07 \times 10^{-5}$ ([6]); thus the change in k from air alone to a mixture of air and CO and finally to CO and CO_2 should readily be discernable by noting the variation in temperature of a red-hot platinum wire spiral surrounded by these gases.

It was realised, however, that in order to avoid poisoning of the heated platinum spirals, arsenical vapours would require removal from the gases examined and finally the apparatus shown in Fig. 1 was devised and fabricated from fused silica, all joints being ground to fit and where possible shellaced over. The reaction crucible A is attached by a short body tube B to the sublimation cap C. Through C an invagination permits the insertion of thermocouple wires. It was not anticipated that much arsenic, either as metal or trioxide would appear beyond C, but to ensure complete absence of arsenicals from the gases to be analysed a short condensing spiral was attached to C and the exit tube lightly plugged with asbestos wool in order to act as a particulate filter. The distal end of this filter tube was attached to the analysis cell E which carried the platinum spiral F.

In operation, two of these assemblies were arranged, one for the charge and the other as a control, the platinum spiral F[1] forming the reference side of the Wheatstone bridge circuit. From Fig. 2, the salient points of the total assembly may be seen. The crucibles A, A′ and body tubes B, B′ were arranged in an electric furnace with the sublimation caps protruding and protectedfrom convection and radiation heating by silvered asbestos sheets. A vacuum set-up was connected via a manometer to the exit tubes from the analysis cells—a slight vacuum of the order of 200 mm. Hg being maintained in order to ensure the passage of the gaseous products over the platinum spirals as soon as possible after formation.

The two identical platinum spirals were connected to a galvanometer G, in shunt via two manganin resistances, dry cells, variable resistance and ammeter as shown, the circuit merely representing an application of the Wheatstone bridge : a change in the rate of cooling of one or the other of the heated spirals altering the resistance of the cell and causing variation in the galvanometer reading. Since the rate of cooling of the spirals depends upon the ambient atmosphere, the change in resistance of a cell as shown by the galvanometer deflection denotes a change in the composition of the gases present. It was realised that no sudden variation in the galvanometer reading would be obtained, but that a more or less sharply defined curve would be manifested on plotting galvanometer deflection against temperature. That this was so is seen in Fig. 3, which indicates the change in composition of an air-CO mixture employed for standardisation. Conjointly with this it was found that the time lag for response of the conductivity cells was negligible insofar as it was apparently of the same order as the lag in recording temperature on the pyrometer.

The carbon employed was 250–300 mesh grain size, wood charcoal of 0·1% ash. The allotropic modifications of As_2O_3 were prepared by fractional sublimation of Analar arsenic trioxide in a closed tube at 400° C.; the octahedral form—subliming to the top of the tube, the monoclinic phase depositing on the central portion

Fig. 1.–Reaction unit.

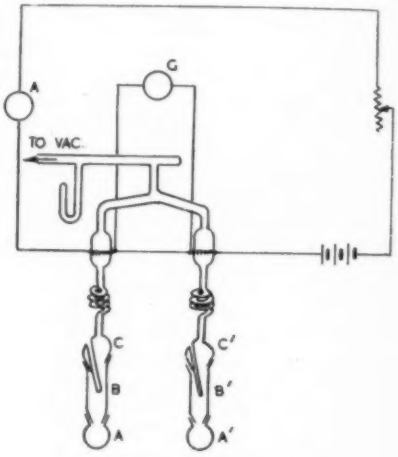

Fig. 2.—Apparatus assembly.

of the tube which was at a temperature of *ca.*200° C., whilst the vitreous, amorphous, phase remained unvolatilised at the bottom of the tube. That it was possible to retain the vitreous phase at 400° C. is due to the increased pressure obtained by this method of preparation. The individual allotropes thus obtained were separately collected, crushed to 250–300 mesh, and stored over magnesium perchlorate.

Experimental

Reaction Temperatures.—The first feature of the reaction which had to be defined was the temperature at which reaction occurred between, or rather in the presence of, the solid phases of each of the allotropes of As_2O_3 and carbon.

Twenty-five grammes of a mixture of a specific allotrope and charcoal in the molecular proportions indicated by equation 1, were fed into the reaction crucible A, the complete assembly adjusted and the furnace esis tance set for a temperature raise of 100° C. in 15 minutes. Galvanometer deflection was then plotted against temperature ; the same procedure was employed

3 *Tr. Farad. Soc.*, August, 1938.
4 *Dict. Appl. Physics*, **5**, 144, 1923.
5 *J. Chem. Soc.*, **125**, 694, 1924.
6 *Hodgeman & Holmes, Hbk. Chem. & Phys., 25th Ed., N.Y.*, 1941.

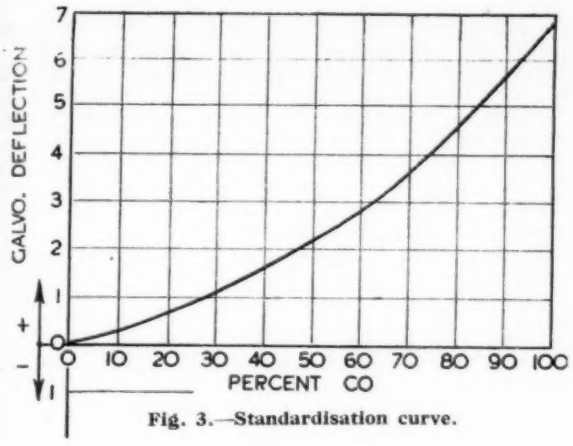

Fig. 3.—Standardisation curve.

for each of the As_2O_3 allotropes. For comparison, 2·08 gm. of charcoal (i.e., the same weight as employed in the previous experiments) were introduced into the reaction crucible and the temperature of oxidation ascertained in the same way as the reaction temperatures previously. The curves obtained are shown in Fig. 4. As is obviously indicated, very little reaction at all occurred with the octahedral allotrope, the CO production curve being almost identical with the carbon oxidation curve. The monoclinic and amorphous phases gave respectively better reactions—the reason for the apparent variance of the activity of the phases is obviously due to the fact that by the time the temperature was reached at which the carbon would react, the allotropes had volatilised and were partially present in the reaction crucible in their vapour phases.

The astonishing feature is that any reaction at all occurred, but that it did is attributed to the differences in specific heat of carbon and the As_2O_3 allotropes, thus: whilst the As_2O_3 particles at the periphery of the charge volatilised readily at their respective vaporisation temperatures, the depletion of As_2O_3 on the outside of the charge left a heat absorbing boundary layer of carbon which, having a lower heat transfer number than arsenic trioxide, was consequently at a higher temperature than the residual As_2O_3 which, remaining at the oxidation temperature of carbon was able to react.

In each experiment the yield of metallic arsenic was determined by weighing the residue and sublimate separately, dissolving out the arsenic oxides with concentrated sodium sulphide solution, and in the case of the sublimate reweighing the residue as metallic arsenic. In the residue from the reaction crucible the insoluble elemental arsenic was determined after Na_2S extraction by solution in concentrated nitric acid followed by iodimetric determination of the arsenic acid so formed. From the sodium sulphide solution arsenic trisulphide was precipitated by acidifying, collected and weighed as usual. The analytical results thus obtained are given below:—

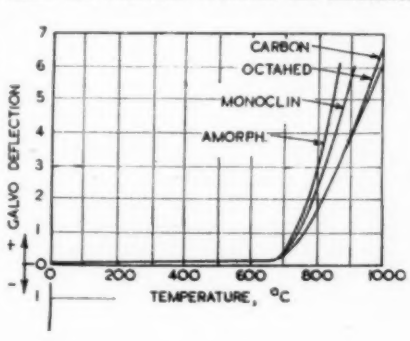

Fig. 4.–Oxidation and Reaction curves.

As_2O_3 phase	Residue		Sublimate		% As yield
	As_2O_3	As	As_2O_3	As	
	%		%	%	
Amorphous	1·5	nil	18·5	81·2	79·5
Monoclinic	0·92	nil	39·0	60·8	59·0
Octahedral	nil	nil	74·3	25·3	24·5

The data thus far obtained indicate conclusively that the temperature of reaction is dependent upon the oxidation temperature of the carbon, but that the yield of As is dependent intrinsically upon the As_2O_3 phase from which it is prepared insofar as upon the volatilisation temperature depends the quantity of As_2O_3 remaining with the carbon until reaction temperature is reached.

Use of less volatile arsenic compounds.—As has been indicated previously, in practice, sodium carbonate had been added to the charge in order to form arsenites and arsenates requiring a higher temperature for volatilisation; such a procedure can obviously only be regarded as of partial benefit since some of the arsenic trioxide will be volatilised before the temperature is reached at which combination between the sodium carbonate and the As_2O_3 occurs, that this is so may be observed from the following data obtained from experiments at 800° C. in which previously prepared sodium arsenite was used: (*a*) and in which sodium arsenite was prepared *in situ* as above (*b*).

	Residue		Sublimate		% As yield
	As_2O_3	As	As_2O_3	As	
	%		%	%	%
(*a*)	1·0	nil	29·7	69·7	65·4
(*b*)	0·6	nil	53·6	45·2	42·1

Similar results would also be encountered in any technique which involved preparation of an alkali or other arsenical *in situ*, consequently the only factors whose determination would be of any value are the respective reaction temperatures of such arsenicals as might be usefully employed. To this end, therefore, the temperatures of reaction with carbon of the following compounds were determined as previously, the reactions assumed to occur being also indicated:—

Basic calcium arsenate	$2\,(CaO)_4As_2O_5 + 5C \rightarrow 8CaO + 4As + 5CO_2$	720° C.
Copper arsenite	$2Cu_3As_2O_5 + 5C \rightarrow 2Cu + 4As + 5CO_2$	690° C.
Magnesium metarsenite	$2\,Mg(AsO_2)_2 + 3C_2 \rightarrow 2\,MgO + 4As + 3CO_2$	725° C.
Potassium hydrogen metarsenite	$2KH\,(AsO_2)_2 + 3C \rightarrow K_2CO_3 + 4As + 2\,CO_2 + H_2O$	700° C.
Copper acetoarsenite	$2\,Cu_4(AsO_2)_6(CH_3COO)_2 + 3C \rightarrow 4\,Cu_2O + 12As + 11CO_2 + 6H_2O$	212° C.
Calcium arsenite	$2Ca_3(AsO_3)_2 + 3C \rightarrow 6\,CaO + 4\,As + 3\,CO_2$	710° C.
Sodium arsenite	$2NaAsO_2 + 2C \rightarrow Na_2CO_3 + 2As + CO$	690° C.

Summarising thus far, the following salient facts are found: The reaction temperature of the components As_2O_3 and carbon depends upon three factors—viz., the temperature of oxidation of the carbon; the vapour pressure of the arsenic trioxide or its compound; the heat transfer values of the components. These three factors may be collated thus: when a mixture of carbon and arsenic trioxide is heated, the arsenic trioxide at the

periphery of the charge is volatilised at a temperature depending upon its allotropic modification until a layer of unmixed carbon of lower heat transfer value remains as a semi-insulating boundary, the temperature of which will be many degrees higher than the core of unvolatilised arsenic trioxide and carbon. When the temperature of this outer shell reaches *ca.*690° C. reaction immediately occurs with the unvolatilised trioxide yielding elemental arsenic vapour which condenses on reaching a cool surface. The amount of arsenic trioxide remaining available for reduction at 690° C. is dependent upon its allotropic form, thus the forms with the higher vapour pressures—octahedral and monoclinic—volatilising as they do at about 400° C. below the reaction temperature give very low yields of elemental arsenic, and even this is somewhat diluted by sublimed trioxide. The allotrope with the higher volatilisation temperature—the amorphous phase—remains unvolatilised for a sufficient time to allow the external carbon shell to reach the reaction temperature, and thus a much better yield is obtained of much purer elemental arsenic.

Retention of arsenic trioxide up to the reaction temperature by conversion into inorganic compounds of lower vapour pressures would appear to be applicable as in some cases are obtained decomposition temperatures higher than the reaction temperature.

Replacement of carbon by carbon monoxide.—From the work recorded in the previous sections, it was evident that reduction of arsenic trioxide by carbon cannot proceed until the latter is present as carbon monoxide. Obviously, therefore, the use of carbon monoxide instead of carbon was indicated if efficient reduction was to be obtained and the reaction of As_2O_3 with CO was, therefore, examined by the methods previously employed with the solid phases. Modification of the reaction crucible was necessary, however—a side tube for entry of CO being attached to the body tube.

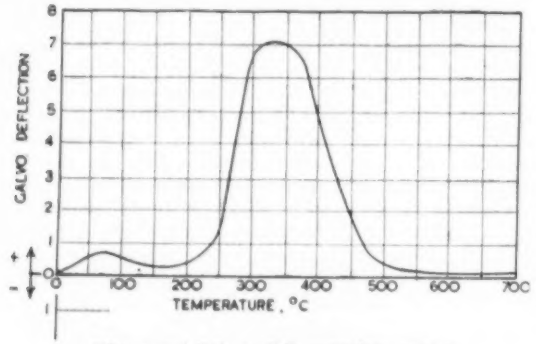

Fig. 5.—As_2O_3 + CO reaction curve.

Carbon monoxide prepared from CO_2 by passage over charcoal at 800° C. and then through caustic soda solution for removal of residual carbon dioxide, was passed through the apparatus at the rate of 1½ litres per minute. With the usual 22·93 gm. As_2O_3 contained in the reaction crucible, the temperature of the furnace was raised from room temperature to 700° C. at the rate of 5° C. per minute. The galvanometer readings of the analysis cells were recorded as in Fig. 5.

The composition of the curve is extremely interesting: the initial decrease in conductivity of the gaseous products of the reaction indicate that an almost immediate reaction occurs between the CO and As_2O_3; after a short time, however, this initial reaction appears to cease—probably owing to the formation on the peripheries of the trioxide particles of non-reactive elemental arsenic. On reaching 200° C., however, rapid reaction at once occurred due to the removal of the elemental arsenic coating by vapourisation. The final increase of conductivity indicated completion of the reaction. Further, it was observed by weighing the elemental arsenic produced that as far as the arsenic trioxide was concerned the reaction was quantitative.

From the carbon monoxide point of view, the quantitativeness of the reaction may be reviewed on the basis of the equation :—

$$As_2O_3 + 3\,CO \rightarrow 2As + 3CO_2$$
198 gms. 67·2 litres.

Thus, the 22·93 grammes As_2O_3 employed are equivalent to 7·8 litres of carbon monoxide. With a temperature raise of 5° C. per minute, 65 minutes were required to reach the end of the reaction at 325° C. as shown by Fig. 5; the total volume of CO employed for the reduction, therefore, was 98 litres or nearly fourteen times theory.

Economically, therefore, the employment of carbon monoxide to replace carbon would not appear encouraging, but against the vast quantity of CO employed must be balanced the greatly increased quantitative yield of As. It would appear, however, that closer examination of the CO—As_2O_3 system would yield results of greater applicability—further work on this subject is being carried out.

Conclusion

It is considered that the results obtained in the series of experiments here recorded indicate the impossibility of obtaining by reduction of arsenic trioxide by carbon, yields of metallic arsenic at all approaching to theory—this is due to the difference in specific heats and vaporisation temperatures of the two components. Replacement of the As_2O_3 by arsenic compounds of lower vapour pressures may have some effect upon the yield—further work on this point is in progress.

The use of carbon monoxide as a reducing agent gave results confirming the indications of Mellor[1] that As_2O_3 is reduced by CO at ca.60° C.—at such low temperatures, however, a protective coating of As appears to be formed on the As_2O_3 particle surfaces; on removal of this protection by volatilisation at higher temperatures reduction appears to be almost instantaneous and complete. In this case, however, quantities of carbon monoxide vastly in excess of theory are required. From this aspect of the reaction further work now in progress is directed towards the use of carbon monoxide at temperatures above the vapourisation points of arsenic and its trioxide—i.e., on reactions between the vapour phases of As_2O_3 and carbon monoxide.

THOSE who are concerned with abrasive problems will be especially interested in a 32-page booklet recently published entitled, "Ni-Hard versus Abrasion." Ni-Hard is a white or chilled-cast iron which has been developed to withstand abrasive wear and this publication deals with both the metallurgical aspects of this material and some practical applications in some industries where it has been extensively adopted. The information given will assist in the solution of many abrasion problems. A copy of this booklet may be obtained from The Mond Nickel Co. Ltd., Grosvenor House, Park Lane, London, W. 1.

Production of Magnesium Alloy Castings

Gravity and Pressure Die-Castings Technique

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Production Metallurgist, Elektron Foundry, Birmingham Aluminium Castings (1903) Co., Ltd.

The outstanding characteristic of the gravity and pressure die-casting processes is the high degree of accuracy with which finished parts may be produced at a relatively fast rate. This accuracy makes possible the reduction and frequently the total elimination of machining operations, while, providing the size and design are suitable and the proper technique is employed, the processes assure a low production cost where a large quantity of castings of the same design is required. Technique applied in the successful production of magnesium alloy die-castings is discussed.

IT is probable that these processes have been discussed so much in recent years, both as talks and through the medium of the technical press, that any introductory remarks may be regarded as superfluous. However, when dealing with a material like magnesium which possesses very definite properties, a review of the reasons underlying the application of the methods is of value, and in order that no confusion shall arise the two processes will be dealt with separately.

Gravity Die Casting

With certain important exceptions this process may be regarded as a logical advance on the sand-casting method of making castings. Indeed, it used to be said that "The Greeks have a name for it," but the most satisfactory designation obtains in America where such equipment is referred to as permanent moulds. Possibly the most important decision to be made in considering a potential die casting is whether or not the size, shape and quantity of the casting lends itself to this particular method. This is important from every point of view and while it must be taken for granted that quantity production is an essential for die castings to function economically, the fact that large quantities of a particular casting are required must not be allowed to influence this very important factor, for the following reasons: (1) All metal dies are expensive due to the necessary machining that has to be performed upon them, naturally this expense will rise steeply with increasing intricacy of the casting shape. (2) The die for a complicated shaped part, which may include severe undercutting, may be so difficult to assemble and part, and collapsible cores take so long to remove, that the output is seriously prejudiced; in this connection the time which must elapse to allow proper solidification to take place may be so long that the production rate is further retarded. (3) It may be impossible to apply necessary feeders and it must be remembered that once a die has been constructed modifications can only be carried out at great expense, so that in the event of poor feeding and resultant micro-porosity time and money will have been wasted. (4) Very large castings may require such a rapid cycle of metal melting production as to overstrain the resources of the die-shop furnaces thereby upsetting the production of the rest of the shop.

It is clear, therefore, that two courses are open; either most detailed consideration of a casting, at present being made in sand, must be undertaken before attempting to make it as a die casting, or the part must be designed from the start as a die casting; conversion from sand to gravity die can rarely have the same chance of success as with the latter case.

Taking the above into consideration the advantages that may be gained from using die castings are: (*a*) They can be cast to closer tolerances and are therefore dimensionally more accurate; (*b*) the surface is smoother; (*c*) production quantities are high, thus resulting in a cheaper casting; (*d*) they have metallurgical advantages by virtue of finer grain size which promotes increases in ultimate tensile strength and elongation of around 20%. Points (*a*) and (*b*) will mean that machining allowances can be reduced, but point (*d*) has an unfortunate aspect, since a fine grain means rapid solidification, which will commence at the die face and proceed inwards, and, unless compensatory liquid metal can provide the necessary feed, patches of porosity will result.

It is, of course, understood that a practical guide of actual wall thicknesses and ± tolerances would be very useful, but it is felt that, although such figures are quoted as a matter of course in much American technical literature, they serve very little useful purpose when the diverse shapes of castings are called to mind. However, the advantages outlined above are quite positive and all the designer has to do is to seek the maximum degree of advantage that can be obtained by using die castings; this is achieved in one way only, consultation with the die foundry experts in the earliest stages of development.

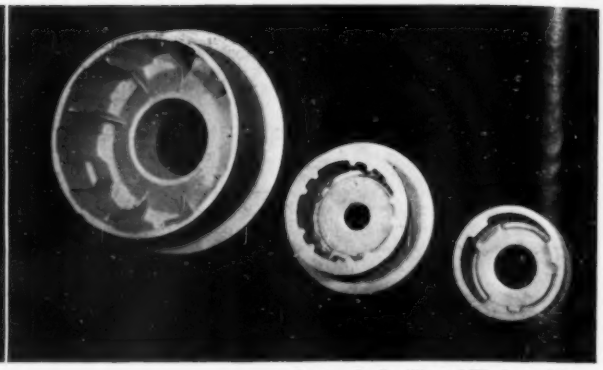
The Die.—It will be noticed that some of the following data applies to the die casting of most metals; this, of course, is inevitable.

Dies are made in a good quality cast iron and are stress relief-annealed before final machining. Their section thickness is varied to ensure correct heat distribution and to encourage feeding, and, as a general rule, a thin sectioned die will work hotter than one with thicker sections.

Cores, due to their constant removal from and insertion into the die, have to resist wear and are therefore made in an alloy steel, usually of the chromium vanadium type; although cast iron cores are quite suitable provided they can stand the shock stresses which may be imposed, coreprint lubrication is provided for by the use of "Aquadag." If undercutting is unavoidable the best practice is to use sand cores, as collapsible steel cores may seriously retard the production time; in the event of sand cores being used, chills are applied where necessary in the same manner as for sand castings. The absence of moisture in a die

Courtesy Dunlop Rim and Wheel Co., Ltd.

Fig. 1.—Die-cast aircraft hubs in Elektron D.T.D. 289. Castings by Sterling Metals.



Courtesy Dunlop Rim and Wheels Co., Ltd.

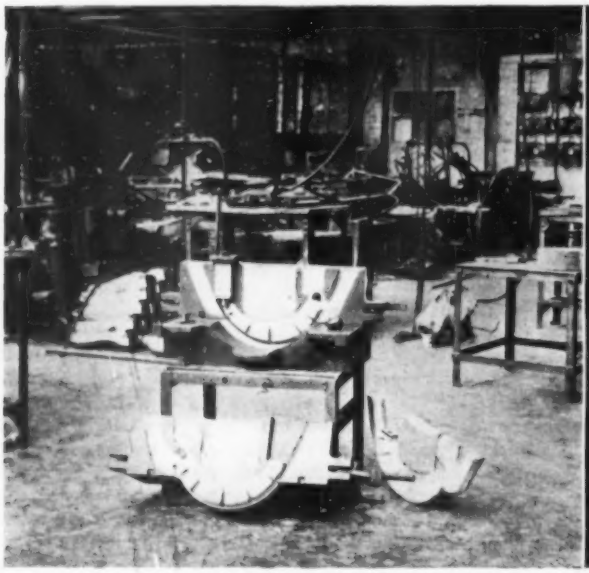
Fig. 2.–Die-cast backing plates for aircraft landing wheels in Elektron D.T.D. 289. Castings by Sterling Metals.

mould allows any sand cores, which may be used, to contain less inhibitor than that usually included in cores for sand castings.

Enough has been said previously to show that, in general, dies must be designed primarily for quick turn-out; all runners and risers are located on the parting line, the die faces of which also contain vents in the form of fine machined grooves. The shape of the runner varies with the size and shape of the casting, but a circular section is avoided owing to risk of a vortex and undesirable features from a practical diemaking standpoint; it is always choked at the ingate area; risers are short and squat, thus resisting the chilling effect of the die a longer time than the casting proper and they are much smaller than those applied to a magnesium sand casting, and are reduced in cross section where they join the casting to provide a witness mark for fettling purposes.

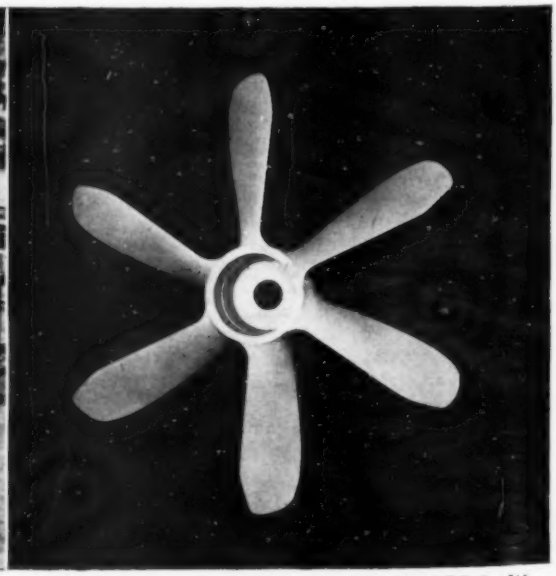
General die design must provide for a runner inducing the least turbulence and the provision of fillets of adequate radii to reduce cracking tendencies; if a sloping runner is impossible to apply tilting of the die can often produce the desired effect. P. Spitaler[4] draws attention to the low specific heat of magnesium based on unit volume, and through this fact, the majority of dies require to be constantly heated by gas jets, while the die is being used, to maintain the correct working temperatures. Although this working temperature of the die is relatively high (referred to later) the magnesium alloy when poured is subjected to considerable chill which, unless allowed for, would prevent many thin walled castings from running and also upset the necessary thermal gradients. This excessive chilling effect is catered for by using a wash or coating upon the die and corefaces; in addition to its insulating effects in this connection it also allows of easy ejection of the casting by preventing it sticking to the die.

A variety of these washes are on the market and they basically consist of whitening or chalk mixed with some boric acid, the whole being mixed with water to a suitable consistency and sprayed on the die when hot; a



Courtesy of Birmingham and Midlands Motor Omnibus Co.

Fig. 3.–Typical die and brakeshoe casting for heavy passenger vehicle in the foundry of Sterling Metals, Ltd.



Courtesy A.E.C. Co., Ltd.

Fig. 4.—Die-cast fan in Elektron AZ91. Castings by Sterling Metals.

binder of the silicate type is sometimes included. With some die shapes the die wash can prove very troublesome and requires frequent retouching; this means that definite rules cannot be laid down, but that the consistency of the wash, temperature of spraying, depth of coat are best decided by experiment; however the life of the coat is prolonged by shaking a fine mixture of talc and boric acid on the die face after each casting is ejected.

The tendency of the metal to oxidise, resulting in high temperatures, may be a factor in causing the coating to

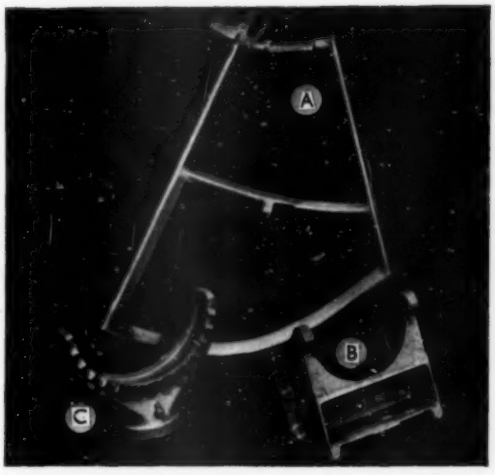
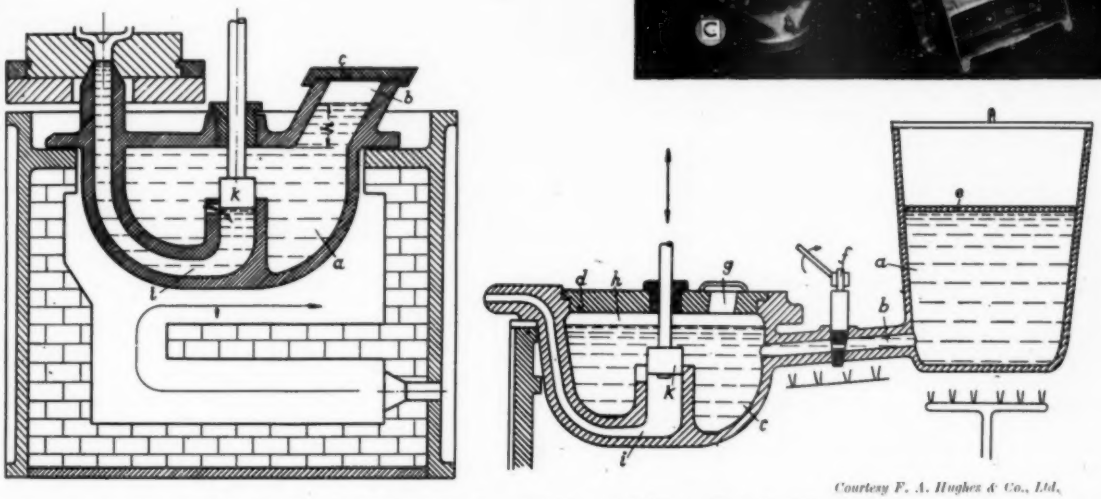


Fig. 5.—Die-cast aircraft components. Castings by Sterling Metals.

(a) Windscreen frame in Elektron D.T.D. 289.
Courtesy A. V. Roe & Co., Ltd.

(b) Control lever box in Elecktron D.T.D. 281.
Courtesy A. V. Roe & Co., Ltd.

(c) Induction belt in Elektron D.T.D. 289.
Courtesy Bristol Aero.



Courtesy F. A. Hughes & Co., Ltd.

Figs. 6 and 7.—Pressure die-casting machines. Hot chamber type.

strip and as an additional precaution sulphur is sprinkled either on the die joint or incorporated in the talc-boric acid mixture just referred to. It is elementary casting technique to spray the risers and runner heavier than the general die face in order that adequate feeding shall be assured, but, as a final refinement and as a complementary help to die design, the coating can be rubbed off locally in spots where more rapid solidification is desired; in such cases the die is rubbed over with a film of graphite to prevent sticking.

Casting Procedure

Metal Preparation.—Owing to its freer running qualities the AZ91 alloy is generally recognised as the most suitable for die castings, but A8 or Elektron "C" material can also be used for many castings if preferred.

Tip axis tilting furnaces of about 160 lb. capacity are usually employed for the die casting of magnesium alloys; units of a larger size being unsuitable as die castings are smaller than the average sand castings; however, a system involving the use of bulk melters to replenish the small pots as and when necessary can be used.

Melting follows the usual practice incorporating "washing" and grain refinement either by superheating or carbon innoculation, the metal subsequently standing ready for use with flux cover. It is at this, the pouring stage where the inherent qualities of magnesium alloys present some difficulty, for it will be recalled that if the flux cover is removed immediate oxidation and burning will take place; this is partly overcome by carefully tilting the furnace and allowing a gentle stream of metal to fill a hand ladle, while flowers of sulphur is continually dusted on the metal surface; this is continued whilst the ladle is carried to the die which is then filled; remedial measures also include the use of the lowest permissible pouring temperatures, and additions of small amounts of calcium of the order of 0·02–0·10% to the pots of prepared metal prior to casting. The addition is made as elementary calcium, which is stirred into the metal a few inches beneath the surface, resulting in an immediate reduction in the tendency of the magnesium to burn; the action is possibly preferential oxidation of the calcium. Pouring temperatures range from 720°–760° C., and as die casting is essentially confined to taking a sequence of small amounts of metal, any one pot will last a relatively long time. With such conditions of time and temperature, coarse grained material is liable to develop which the chilling action of the die utterly fails to cure; the solution either lies in emptying the pot of prepared metal fairly quickly by allocating several dies to the one pot or refining the grain again after a predecided time limit has expired.

Die temperatures are important, and in general the highest possible working temperature yields the best

results, the usual range being from 300° C. to about 340° C., and pronounced hot cracking of castings results from the use of too low a die temperature.

Defects usually associated with die castings are cracks, microporosity, and oxide inclusions, and inspection procedure is much the same as that already described, with reference to cracks, however, extra special means are usually taken for their detection. One method consists of painting on a spirit soluble dye which concentrates in the crack and is shown up visually on drying out.

Microporosity has already been discussed and is present in die castings if the balance between feed and chill is incorrect; it is probably the most common defect encountered in the manufacture of magnesium die castings. Inclusions of oxide skins occur; if present as black or brown specks, they are introduced through careless handling of the liquid metal in the pouring ladle allowing oxidation to take place.

The principles of good design should be applied to all types of casting, but with magnesium die castings such principles are imperitive. They include the provision of generous radii, adequate fillets and smooth transition in changes of section; cracks will occur if the above are not provided.

The quantities which are usually produced from any one die vary according to the intricacy of the casting, but if a die can be worked until its useful life is exhausted obviously the best economic conditions are obtained. This can mean a total of 25,000 castings, but in general, orders calling for several thousand castings are necessary to enable the method to function successfully.

Pressure Die Casting

At the present time when manufactured parts are increasingly subjected to competition of one sort or another there is an intensive search for faster and cheaper methods of production, and one of the fundamentals of the pressure die-casting process is its high speed of production compared with the sand and permanent mould methods.

Coupled with the knowledge that an expensive machine and die forms part of the equipment, it follows that due to the cost the method is essentially applicable to the production of large quantities whether or not the requirements of the desired casting fulfill those enumerated below. Usual quantities necessary for both the manufacturer and customer to derive maximum benefit are from 10,000 to 50,000, but economically this depends upon the type and design of each particular job. The most successful exponents of the art to date (as far as magnesium alloys are concerned) are the Germans and, from published literature on the subject,[1,2] the Americans also appear to be making rapid strides; the process is known simply as die casting in the United States. It is an unfortunate fact that in this country the same advantage of the process has not been taken, although large numbers of various types of casting are produced from the zinc-base and aluminium-base alloys. When it is realised that each country has independently arrived at what is nominally the same alloy, for actual compositions, see Table I, the above observations are brought into greater significance, and these analyses tend to show that the ability to make pressure die castings in magnesium alloy successfully hardly depends upon the material as such or the melting procedure involved for it is recognised that with such an easily oxidised metal special needs arise.

TABLE I.—MAGNESIUM PRESSURE DIE-CASTING ALLOY COMPOSITIONS.

English	American	German
Al% .. 9·0 –9·5	9·0	9·30
Zn% .. 0·45–0·60	0·6	0·40
Mn% .. 0·20	0·20	0·15
Si% .. 0·10–0·60	1·00 Max.	0·21
Fe% .. 0·02 Max.		0·03
Ni% .. Trace	0·01 Max. } 0·3 Max.	Trace
Cu% .. 0·04	0·05 Max. }	0·11
Pb% .. 0·02		0·02
Sn% .. 0·03		0·06
Mg% .. R.	R.	R.

The reasons which appear to have exercised a restrictive influence upon the expansion of the magnesium pressure die-casting industry in this country are: (1) The material by virtue of its inherent oxidisable qualities calls for special handling methods which increase the cost of production and render the general operation more difficult. (2) The behaviour of the alloy during solidification is not fully appreciated, and dies are allowed to be put into work which overstep the limits of permissable design. It can be mentioned here that work has been carried out using an alloy of an entirely different composition to that mentioned in Table I with superior results.[3] Although more work remains to be done the results obtained tend to prove this point. (3) The suitability of magnesium for certain parts has not been appreciated by consumers, with the result that the large quantity orders necessary to stimulate the process have not been forthcoming.

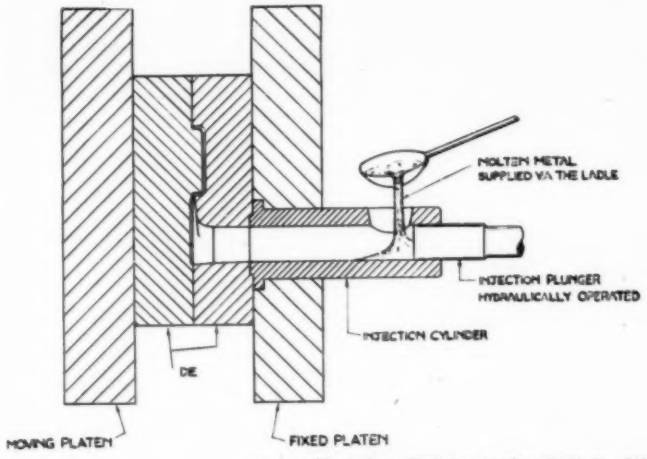
The correctness or otherwise of the above does not influence the fact that the wonderful castings which are made in Germany from nominally the same material as is used here are not called for in this country. However, in fairness it is necessary to delve somewhat deeper into the why and wherefore, and trust that the true explanation will emerge. It must be borne in mind that the political situation in Germany demanded a large degree of national self sufficiency and magnesium alloys satisfied this need, undoubtedly this proved a major stimulant to the industry. Also the quality of many German castings leaves much to be desired not with regard to surface finish but from the standpoint of internal soundness and oxide inclusions and British manufacturers in general hope to produce something better. Special attention should be directed to reason (1) enumerated above for the difficulties encountered in the process lower the economic efficiency considerably, high melting loss is a case in point and remains a major problem.

Design Principles and General Characteristics.—An excellent article already referred to mainly dealing with the dimensional side of the process leaves little to be desired on this subject.

The greatest trouble likely to be encountered when using the process is the prevalence of cracks. Cracked castings are the bane of the magnesium pressure die caster and design should be directed to the elimination of this defect. In general the usual formulæ comprising the use of large fillets, uniform section, gentle changes of section, and the rounding of sharp corners, must be followed, but, in addition, there is the very important consideration of draft angles or taper which, unless of a very adequate nature, will prevent the rapid ejection of the casting from the die. This is very important from the point of view of speed and cracking, for contraction on to inside faces will favour crack formation and the setting period is so short that almost immediate ejection is necessary.

The particular advantages which are possessed by castings made by this process are: (*a*) The process allows of the production of exact

1 Powe and Scott. *Product Engineering.* October, 1945.
2 George B. Hansberg *Die Casting.* November, 1946.
3 Birmid Internal Reports.



Courtesy Birmingham Aluminium Castings (1903) Co., Ltd.

Fig. 8.—Diagrammatic part section through a "cold chamber" pressure-casting machine.

duplicates at low cost. (*b*) Extreme dimensional accuracy is obtained. (*c*) The castings have a very good finish which incidentally comprises a tough skin exceptionally resistant to corrosion. (*d*) Machining operations are reduced to a minimum and the high speed of production has already been mentioned.

The introduction of pressure-die methods has widened the casting field in that thin walled and complicated castings can be produced; indeed, thick sections and excessive bosses are to be discouraged as they are liable to induce zones of porosity. The low tensile strength of the material at high temperatures combined with the fact that the section thickness is small emphasises the need for caution in design of fillets to reduce the tendency towards cracking.

The size and weight of castings are limited by the casting machine available, but Powell and Scott refer to approximate maximum dimensional limits as 8 in. deep × 12 in. wide × 15 in. long, they also include data on wall thickness and manufacturing tolerances, but these detailed points are best discussed with the foundry experts when a potential pressure die job is mooted; if the shape of the proposed casting is suitable the user may find that the founders can give him closer tolerances than he expects. However, to further emphasise this desirability of co-operation it must be remembered that magnesium pressure die castings are cast at a much higher temperature than zinc-base alloys and therefore cannot be held to the same tolerance, on the other hand, as no chemical attack between the alloy and the steel die occurs they are made as accurately as aluminium base castings. Any necessary extra strength is provided for by designing with ribs, and cored holes are easily accommodated, but the maximum advantage will be gained by designing with stationary cores—i.e., those which are integral with the die halves and are therefore automatically decored on opening the die; this particular aspect results in considerable saving in machining. The practical disadvantages of the method are the difficulties associated with the handling of the molten magnesium alloy, and the fact that the die temperature must be kept up. The handling difficulties cause high metal losses by oxidation and also introduce the risk of the resultant casting containing inclusions of oxide and particles of flux.

The Die and Cores.—The useful life of the die is determined by the casting tolerances allowed, and when these can no longer be held it must be discarded. Fortunately as no chemical action occurs between the magnesium alloy and the die face, wear is less than that taking place when aluminium-base alloys are used and one die can produce as many as 500,000 castings, but the usual quantities are nearer those previously indicated; the size and intricacy of the design also play their part in determining die life. An alloy steel of the chromium-tungsten type or other special composition is used for all parts of the die in contact with the liquid metal and it is specially hardened and tempered to a Brinell of about 450 to give maximum resistance to wear. Venting is carried out in the same manner as that applied to permanent moulds by machined grooves on the parting line. The working temperature of the die must be maintained. In this connection it is necessary to strongly preheat with a gas flame or other device before starting work, a die temperature of approximately 250° C. is desirable, and failure to maintain this temperature is the greatest contributor to cracking troubles excluding design factors. The close dimensional limits demanded of pressure die castings do not allow of any wash being sprayed on the die faces; the practice of swabbing with a lubricant is most undesirable and should be avoided.

The Machine.—This consists of the apparatus for transmitting the metal from its pot or reservoir to the die gate and also includes the die parting

Courtesy Birmingham Aluminium Castings (1903) Co., Ltd.

Fig. 9. Cold chamber machine in operation.

and closing mechanism. Machines are usually of two types: (*a*) Those which contain either the melting pot or a large holding pot of metal usually known as the hot chamber or plunger machine and; (*b*) those in which the reservoir of metal is separate from the machine and the necessary amount is charged in by hand ladle, this is known as the cold-chamber process.

Fig. 6 and 7[4] are drawings of typical German hot-chamber machines, and a brief description follows. Fig. 6 shows the liquid metal reservoir "a" which is closed from the air except for the feeding shaft "b" through which new ingot metal is fed, oxidation is retarded by the addition of pinches of sulphur from time to time through the same opening. The castings are made by injecting the metal into the die at pressures of approximately 1,500 lb./sq. in. by means of the steel piston "K" operating downstrokes in the pressure container "i," the port allowing metal to enter the pressure chamber is automatically closed by the piston "K" during each stroke.

The machine sectioned in Fig. 7 differs from the former only in that there is a refinement in the metal handling equipment, such machines are fast workers and these or similar types are still believed to be popular to-day. The machines are mainly hydraulically operated and metal temperatures are usually of the order of 650°–670° C.

The cold-chamber machine, shown in Fig. 8, is used for aluminium as well as for magnesium alloy. A ladle of metal is taken from a pot near at hand, poured into the cylinder immediately by depressing a foot pedal whereupon the metal is forced into the die cavity at a pressure ranging from 8,000-lb./sq. in. to 25,000 lb./sq. in. at a temperature of about 620° C.; this type is also operated hydraulically but is not so fast as the plunger machines.

4 A. Beck. "The Technology of Magnesium and Its Alloys."

Treatment of Metal

The melting procedure follows the normal practice of cleansing and grain refining, but the problem which has to be solved is that of preventing the burning of the metal during its manipulation from the reservoir to the die-casting machine. Some of the methods which have been tried to overcome this difficulty have already been mentioned, and they consist basically of an attempt to maintain a layer of SO_2 or burning sulphur over the liquid bath. This would be an excellent method if it were completely efficient; in spite of all precautions, however, even at temperatures as low as 620° C., some drossing up persists with the consequent formation of particles of oxide and nitride which float in the body of the melt, contamination of the casting automatically follows. It must be made clear that the above systems, although possessing these drawbacks, are used on a large scale and have been responsible for the production of many castings. In the majority of cases some small inclusions can be tolerated, since, generally, pressure die castings are not used for stressed parts.

For the hot-chamber process it seems certain that the best method is for the pot of metal to be protected in the normal manner by a flux cover, the metal required for "shots" being taken from the body of the melt, in this way oxide free and flux free metal is obtained, but the difficulty of this method is the necessity to obtain careful additions of solid ingot so that no flux is carried under.

With regard to the cold-chamber process it is possible to take a ladle of metal containing enough for one "shot" by dipping into a normally prepared pot of flux-covered metal (it is not possible to carefully pour off as in permanent mould casting owing to the speed of the process, small quantity of metal required, and low temperatures used). In spite of the dexterity of operation obtained by long experience this method is prone to flux inclusions and is unsatisfactory from this point of view.

Latest attempts to deal with the oxidation problem have consisted of making small additions to the alloy which reduce its oxidation tendencies and the most important element used up to the present is beryllium. It is now known that beryllium has remarkable properties in this respect, additions as low as 0·006% visually decreasing the burning of the alloy. It is added usually in the form of a Be/Al hardener and similarly to the way calcium is added to die-casting pots (previously described). It seems highly probable that this approach to the problem will increase in importance, because, if the metal can be rendered quiescent by such simple means, a serious working obstacle has been removed. There is one drawback to beryllium addition and that is the very large increase in grain size of the magnesium alloy which occurs even with very minor amounts, this is not too serious providing that beryllium containing scrap is kept strictly for pressure die use, it would be disastrous, however, if allowed to get into the sand foundry.

Magnet Steels and Alloys

THERE has been an enormous increase in the use of permanent magnets, in the last two decades, for new applications which have sprung from the rapid growth of the radio and other electrical industries. Demands for improved magnet materials have stimulated research which has led to the development of new steels and alloys with greatly enhanced mechanical properties permitting far-reaching economies both in weight and cost. The more recent achievements in this field have been made by British scientific work and in this the firms of Jessop and Saville have been particularly prominent. There is evidence that as a result of continuous investigation new permanent magnet materials will continue to be developed, but the range of materials described in a new booklet on the subject by William Jessop & Sons, Ltd., and J. J. Saville & Co., Ltd., indicates the great progress already made. Copies are available on application to Wm. Jessop & Sons, Ltd., Brightside Works, Sheffield.

The Iron and Steel Institute

THE annual general meeting of the above Institute will be held on May 14th to 16th inclusive. The technical discussions on May 14th will be held at the offices of the Institute, 4, Grosvenor Gardens, and those on May 15th and 16th in the lecture theatre of The Institution of Civil Engineers, Great George Street, London, S.W. 1. There will be a dinner for members and their guests at the Connaught Rooms, Great Queen Street, W.C. 2 on May 15th, at 7-15 p.m.

AT a recent meeting of the Council of the British Iron and Steel Research Association, Mr. G. H. Latham was appointed President in succession to Sir James Lithgow, Bt., G.B.E., M.C., the first President, who was elected in 1945 when the association was formed. Mr. Latham, who is President-elect of the British Iron and Steel Federation, and a member of the Steel Board, is Chairman and Managing Director of the Whitehead Iron and Steel Co., Ltd., Newport.

The British Industries Fair

The world's greatest national trade fair opened on May 5 in three great buildings at Olympia and Earls Court in London, and Castle Bromwich, Birmingham, and will remain open until May 16. The Fair reveals to the home buyer, as well as to the overseas buyer, the swiftness of Britain's industrial reconversion, the technical advances gained from wartime experience, and the vigour with which industry has applied itself to export demands. Here an effort is made to review the more salient features of the exhibits which come within our field and is primarily concerned with those displayed at the Castle Bromwich section.

THE British Industries Fair, which opened simultaneously in London and Birmingham on May 5 and will remain open until May 16, was held annually from 1915 until 1939 and had become recognised as one of the most important commercial events of the year. It was necessarily suspended during the recent war, thus it is eight years since the last Fair was held, but its revival had been under consideration for the last four years, and a recommendation made that it should be organised to resume in 1947. Having given this advice, the Committee responsible for it felt that their period of usefulness was finished; they felt that the period of office for which they were elected had lapsed during the war. It is noteworthy, however, that the confidence of the members of this Committee in the successful conclusion of hostilities and the recovery of British industry was of such a high order as to warrant this year for the revival of the Fair. That their foresight and courage was not misplaced shows how well they understood the spirit of the nation. It was recommended that the new British Industries Fair should be planned to be widely representative of United Kingdom industry and thus make a maximum contribution to post-war export trade.

Despite difficulties which have had and are having a great effect on Britain's industrial production, and which at one time were thought to be so critical as to cause postponement of the Fair, a record number of 3,131 manufacturers are participating in this the greatest national Fair ever held. At Olympia there are 1,287 exhibitors, and at Earls Court 866, making a total of 2,153 in the London section, where the lighter industries are exhibiting. At Castle Bromwich, where the engineering and hardware section is housed, 978 manufacturers are represented.

After a lapse of eight years a Trade Fair serves a far wider purpose than the immediate placing of orders. It gives the overseas buyer an opportunity to consider Britain's progress in relation to parallel difficulties throughout the world. He can discuss these problems with the actual manufacturer. He will renew old friendships and make fresh contacts while reviewing hundreds of unique advances in industrial technique which have resulted from experience gained in war. In addition, at this Fair, buyers can inform themselves and prepare for sound business decisions in the years when quantities will increase and buying policy will depend on advance knowledge.

In dealing with such a comprehensive Exhibition, where so many technical advances are displayed, much more space would be needed, even if they were presented in catalogue form, than can be given here. Under the circumstances, therefore, it is probable that a general outline of progress, which has and is taking place in the iron and steel and non-ferrous industries in this country, would serve a useful purpose in assisting visitors to the Fair to understand some of Britain's basic industries. Progress reports of a number of basic materials in the field of this journal were given in the December, 1946, issue and the following notes can be regarded as supplementary.

British Steel

The technical advances in the steel industry had an early start in this country, where many of the pioneers, like Cort, Huntsman, Bessemer, Thomas, Siemens and others, carried out developments that provided the basic knowledge on which the steel industries are built throughout the world. Progress has continued through the years in an endeavour to meet the increasing service needs of engineers, and to-day British steel, in regard to quality and reliability, is as renowned as in earlier days. Technical advancement is, of course, intimately bound up with different basic problems, which very often influence fundamentally the economic aspect.

Steel occupies what is essentially an intermediate position in the national economy. On the one hand it draws on the great raw material sources—coal mining, iron ore mining, collection of scrap and, of course, makes heavy demands on the transport system for assembling vast quantities of these raw materials as well as of many other materials—limestone, refractories, etc. Then at the finishing end it provides the principal raw materials for very many of the major industries—shipbuilding, automobiles, engineering. It plays a great part in agriculture and an increasing part in house building, particularly in the provision of household equipment, and to an increasing extent in mining and other activities. It plays an important part in the export trade which is so vital to this country, and any increase necessary in present circumstances must come mainly from the steel and steel using industries. Already in 1946, 42% of our exports depended on steel—10% in the form of direct export of steel products and 32% in the form of the products of the steel consuming industries.

This central position of the steel industry in our economy makes it a particularly good index of the degree of industrialisation of a country. Thus, the consumption of steel per head of the population in America is 860 lb. a year, in the U.K., 520 lb; in Australia, 470 lb.; in France, 260 lb.; in India, 8 lb.; and in China, 5 lb. These figures give some idea of the great possibilities of industrial development that exist in many parts of the world.

In another sense the steel industry occupies a key position in that fluctuations of steel output provide a good and sensitive index of the welfare of the country's economy. The ease with which capital development and purchases of durable goods can be postponed when the trade outlook is unfavourable means that the demand for steel falls away rapidly in periods of bad trade. Thus, in five out of twenty years between the two world wars, the output of steel fell below 50% of the capacity of the industry. Recently successive British Governments have devoted a great deal of thought to the problem of ensuring much greater stability of employment and production in this country. It is generally accepted that the main key to the achievement of a high level of employment is to maintain a stable demand for capital equipment. This is a difficult problem and involves policies far beyond the field of the steel industry alone. Nevertheless, the steel industry in formulating a development plan for the next five years has shown the sort of forward planning of capital expenditure which, if it can be developed over a wider field, will go a long way to providing an overall stability in the item of national expenditure which was previously most subject to extreme fluctuations.

This was based on the scrapping and re-building of about 40% of the existing capacity of the industry, and expanding its capacity overall from about 14 million tons a year to 16 million tons. The total cost of the scheme was estimated at £170 million. This plan of development has already been started on and the benefits will begin to be felt during the course of 1947 from some of the minor schemes which can be quickly completed. The major developments, however, must take some three or more years to complete.

Possibly the fundamental economic factors in any steel industry are those determining its location. In this respect, Great Britain has considerable natural advantages. It has the three main raw materials available—coal, iron ore and scrap—and it has big markets close to the centres of production and a highly skilled labour force; the total numbers employed in the industry being about 300,000. Of these raw materials, scrap is relatively in more abundant supply than in any other steelmaking country, good quality coal is normally abundant, but iron ore available for working in large quantities in the U.K. is of relatively low iron content, containing only 30% iron. These home ore supplies are, therefore, supplemented by the importation of higher-grade ores from Sweden, North Africa and other sources.

No account of the steel industry, however brief, would be complete without a reference to the excellent relations traditionally obtaining between labour and management. The steel industry is one with a long tradition of craftsmanship, and this has led to a deep-rooted pride in the achievements of the industry and a sense of community of interest between management and workers. The importance of this tradition is shown by the fact that no strike has originated in the industry during the last 50 years. Moreover, the steel industry is traditionally one where wages and output are directly linked. The basis of most of the wage scales in the industry is the tonnage rate per unit of output, so that the community of interest in maintaining quality of output is also present in maintaining quantity, to the mutual advantage of both workers and firms. The latest and most striking example of the good labour relations is the agreement between the two sides of the industry by which all melting plants, where it is technically possible, will work a continuous seven-day plant week with a 48-hour week for the men on a shift system instead of closing the steel furnaces down at week-ends. This continuous week is expected to have many advantages, including an increase in output per unit of plant, and stands in striking contrast to the reduction in the working of plant in some other industries.

The problem of the relation between the State and the Industry is of particular significance. On the one hand the State has entered directly into the operation of many of the public utility services of the country. Equally, at the other end of small scale manufacturing and distributive businesses, it is quite impossible to visualise any detailed operation of such units by the State. In the intermediate field of the Steel Industry, however, the issue is a very real one. Over a period of years co-operative action within the industry has become of increasing importance. As the size of unit has become progressively larger, competition has ceased to be the sole factor governing the development of the industry, and a growing factor in that development has been co-operative planning between firms. This has been an important consideration leading to the view in some quarters that the whole industry might be taken over by the State. There are grave difficulties here, arising partly from the fact that the production of steel is so closely associated with manipulation into a great variety of forms and products so that there is no clear dividing line between the steel industry and the industries concerned with the production of metal products.

As an immediate step there was set up by the Government in the Autumn of last year an Iron and Steel Control Board containing representatives of management, workers, consumers and the Government, and this body has complete authority over prices, development and other matters of policy connected with the industry. It, however, exercises only a supervisory function in relation to production, leaving to the firms the financial risk and the responsibility for the actual organisation of production. This arrangement provides a machine under which public control can be operated to ensure that the policies of the industry are in line with national needs; while at the same time it does not represent such a degree of centralisation as will destroy the initiative, the incentive and responsibility of the management of the individual firms.

Technical developments have proceeded at a rapid pace, and there is a tendency towards a return to the use of higher alloy types of steel to ensure a more uniform and reliable heat-treated product, but it is likely that, for economic reasons, austerity will continue to be applied in the use of alloying elements. However, special purpose alloys, such as those for magnets, high-temperature alloys, etc., have been developed which are outstanding. For instance, forgeable permanent magnet alloys are now available which are comparable in performance with the best cast alloys, which have about three times the energy content of good alloys available before the war. In the development of the gas turbine materials have been developed which permit operation at a temperature in the neighbourhood of 700°–750° C. Development of new alloys is proceeding which will probably raise the operating temperature ultimately to 850° C.

The British Copper Industry

Like so many British industries essential to the war effort, that concerned with copper and its alloys has felt the effects of two mutually opposing forces, during the years of the struggle and in the aftermath which had followed. An urgent dynamic stimulus for the development of new alloys, improved processes and accelerated methods of production to meet the exigencies of war has been curbed by the difficulties of obtaining materials, shortage of man-power in industry and, for most of the time, blackout and bombing. Both these factors will be discernible in the notes which follow, typical instances of each being, respectively, the development of free-machining copper, so essential to radar in its earlier forms, and the shortage of tin which led to the unavoidable substitution of lower grade materials for the tin-rich bronzes and gun metals.

To describe in detail the major war-time trends of the British copper industry would be trite. It is obvious, for instance, that vast tonnages of cartridge brass were fabricated into cartridge cases, whose priming caps were made of cap metal, that every projectile of any size was fitted with bands of copper, that bronze, gun metal and high tensile brass castings formed essential parts of ordnance, aircraft and transport by sea and land, that cupro-nickel and aluminium brass condenser tubes helped to maintain our ships and power stations, while copper itself provided the basis of most electrical transmission and equipment. It is the present purpose, rather, to trace certain newer developments which have arisen during, and largely because of the war.

It is well known that the addition of lead to brasses, especially those of the Muntz metal type, improves their machineability. The same is true of the addition of lead to copper, but the hot workability of the copper is thereby impaired. This is a serious drawback because most copper products are hot worked in the early stages of manufacture. Selenium and tellurium when added to copper, however, greatly improve its machineability without detriment to the hot-working properties. In America the use of selenium appears to be favoured, but in Britain the development of free-machining copper containing about 0·5% of tellurium represents a typical war-time advance. The early forms of the magnetron, the very "heart" of radar equipment, demanded a material of high electrical conductivity which could be machined to exceptionally fine tolerances. It is probably true to say that had it not been for the availability of tellurium copper the development of the magnetron, in which Britain claims a pioneering lead, would have been seriously retarded.

The need for high strength parts of intricate shape and good resistance to corrosion for ordnance and aircraft, caused a very large expansion in the production of aluminium bronze castings and forgings during the war. Though straight 10% aluminium bronzes are frequently used, those containing iron are more popular, and for purposes where exceptionally high strength and resistance to wear are required the alloy containing 10% of aluminium with 5% each of iron and nickel is greatly favoured. Aluminium bronzes are readily hot forged, particularly so because they can be heated without fear of spoiling their surface by oxidation, though somewhat more powerful presses are required than for brass and copper. On the other hand, casting and welding present certain difficulties, and in these directions British industrial technique has made considerable advances. Large quantities of both sand and gravity die castings of excellent quality were produced in aluminium bronze for armament purposes. The welding of aluminium bronze, especially by arc methods using coated electrodes, is rapidly passing from the development stage into normal industrial practice.

Three other copper-base alloys which have felt the stimulus of the war, and which are now much more widely used than before 1939 are copper-lead for bearings, chromium copper for welding electrodes and similar purposes and beryllium copper, with or without cobalt, nickel and other elements, for the high precision springs of electrical and scientific instruments. Copper-lead, as a bearing metal for internal combustion engines and all kinds of high duty purposes, has undoubtedly come to stay, and all the largest manufacturers of bearings in the country are actively engaged in its production. A certain amount of reticence is observed in regard to technique, but powder metallurgy, electrolytic and casting methods are in use. Additions of such elements as tin, nickel and silver are common, the usual lead content ranging from about 25–40%. The bearing metal is generally supported upon steel strip and formed to size by press-work followed by accurate machining.

At one time, chromium copper was advocated for the cylinder heads and similar parts of air cooled internal combustion engines, owing to its good thermal conductivity combined with high strength in the heat treated condition. At present, however, it is used mainly for the electrodes of spot welding and seam welding machines, for which purpose it now ranks with cadmium copper as one of the most popular materials.

Great expansion has occurred in the demand for beryllium copper in spite of its high cost. Mainly it is used for the precision springs of delicate measuring instruments, for the springs of electrical contacts and similar purposes, but quite massive spot-welding electrodes are on the market in cast and heat treated beryllium copper for purposes requiring the application of high pressures. It should be noted that industrial developments in Britain are in active progress regarding the new copper nickel-manganese alloys, which have properties comparable with those of beryllium copper, but which should prove to be considerably less expensive.

Until recent years, bronzes containing upwards of 10% of tin have been used exclusively in the form of castings. A modern development is the production of high tin bronzes in the form of wrought rods and other shapes. The cast billets are first heat-treated to bring all or most of the tin-rich constituent into solid solution in the copper-rich matrix, after which they may be rolled and otherwise worked with comparative ease. They combine good strength and ductility with high resistance to corrosion.

Great advances have been made in the technique of melting and casting of tin bronzes, based upon the researches of the British Non-Ferrous Metals Research Association and of the Tin Research Institute. The improved soundness obtained by oxidizing the melt to eliminate hydrogen followed by adequate reduction with phosphorus or other deoxidant, is now widely recognised throughout the industry. During the period of greatest tin shortage, Britain like her Allies, was faced with the necessity of reducing the tin content of bronzes and gun metals for many purposes, and in some cases of substituting high tensile brasses for them. Much valuable experience was gained, and the fact that there is likely

to be a sustained demand for bronzes and gun metals containing less than 10% tin is reflected by the inclusion of specifications for them in the comprehensive schedule of copper alloy castings now in preparation by the British Standards Institution.

Prolonged investigations by the British Non-Ferrous Metals Research Association have led to the development of cupro-nickels containing a little iron but very much less than the normal 30% of nickel for condenser tubes in marine engineering. These alloys are especially resistant to impingement attack by aereated sea water, and like other cupro-nickels, are easy to fabricate. The reduction of nickel content marks an economy in cost.

Among the processes as distinct from alloys, which were developed to meet war-time needs, may be mentioned the silver brazing of the honeycombs of copper radiators and intercoolers, especially for aircraft. The parts to be joined are coated with silver, before being set up in a jig in which they are heated to a temperature between the melting points of silver and copper. The impact extrusion, in the cold, of thin-walled copper tubes for radiators was newly used in this country though the process cannot be regarded as a British development.

Courtesy of Reynolds Tube Co. Ltd.

Gas cylinder and sphere in aluminium alloy for high pressures. Note also developments in plate manipulation.

The British copper industry is working to the height of its capacity, and is flooded with orders to such an extent that protracted deliveries are unavoidable. It is, however, according the highest priority to the widespread demand for copper for roofing, flashings and piping to meet the present housing drive.

The production and fabrication of copper and its alloys are illustrated on the stand of the Copper Development Association, where applications in the various branches of engineering, and in the building and chemical industry are shown. The metallurgical section, in particular, will illustrate mining, smelting and refining, and such processes as rolling, wire and tube drawing, extrusion, casting and hot and cold forming. The exhibits will include "native" copper, copper ores (including malachite, azurite, chalcopyrite, covellite and chalcocite), crushed and roasted ore, flotation concentrate, matte, cement copper (prepared by precipitation on scrap iron from a solution of copper sulphate), and many examples of different semi-fabricated forms.

Development in the Malleable Industry

For many years blackheart malleable iron has been finding steady favour in this country. Due to the considerably increased demand for this material for war purposes, producers found the necessity for improving and developing their melting facilities.

Generally speaking the quality of British malleable iron castings has improved over the war years, and a revision of the present British Standard is long overdue. With the development of more modern melting and annealing technique, together with a better understanding of the process of graphitisation, there has been a marked improvement in the mechanical properties of blackheart malleable iron, and most of the well-established producers of this material have little difficulty in obtaining a tensile strength of 22/24 tons/sq. in., and elongation figures of 14–18%. Furthermore, the old idea that the strength of malleable iron lies in its skin can be disproved by the fact that good mechanical properties are maintained after machining.

In addition to the upward trend of blackheart malleable iron, the more forward members of the industry are giving attention to the development of pearlitic malleable iron. This material has a definite future, due to its increased mechanical properties. The tensile strength is comparable with mild steel. The chief mechanical properties of this material are shown below, from which some of the numerous applications of the material can be readily appreciated :—

Ultimate tensile strength, 33–35 tons/sq. in.
Yield strength, 20–22 tons/sq. in.
Elongation, 6–8%.
Brinell, 175.
Bend, 90°.

Unlike most other high-duty materials, pearlitic malleable iron has extremely good machineability, cutting bright and freely. An example of machining a pearlitic malleable casting, produced by one of the leading blackheart malleable iron founders, emphasises this important feature. The job in question was a rear-axle casting machined with a "Stag Major" tipped tool and machined dry. The machining speed attained was 127 ft. per minute, with a $\frac{1}{8}$ in. cut. As in the case of blackheart malleable iron, though not to the same extent, the fine graphite particles dispersed throughout the whole of the section tend to act as a lubricant to the tool.

From the designer's point of view, the most salient feature of pearlitic malleable iron is its high yield strength which ranges from 20–22 tons/sq. in. The comparative yield strength of other materials is as follows :—

Grey cast iron : Cannot be recorded, owing to absence of ductility.

Wrought iron : Approximately 12 tons/sq. in.

Dead mild steel: Approximately 11/14 tons/sq. in.

Ordinary mild steel: 14/21 tons/sq. in. (varying with carbon content).

Blackheart malleable iron: Approximately 13/16 tons/sq. in.

Whiteheart malleable iron: Approximately 12/18 tons/sq. in. (varying with casting thickness).

Pearlitic malleable iron: Approximately 20/22 tons/sq. in.

Pearlitic malleable iron, due to its chemical composition, is suitable for producing castings of heavy section, as the possibility of the development of primary graphite is more remote. Castings up to 6 cwt. each have been made in this material with section up to 4 in. thick. A casting of this weight is generally beyond the normal productive range, but this example serves to indicate its suitability for heavy casting production. Due to the structural and mechanical properties of pearlitic malleable iron, subsequent heat-treatment can be carried out both selectively and locally. Development work is still proceeding in this direction to ascertain the best methods of heat-treatment both in regard to hardening and tempering.

Briefly reviewing pearlitic malleable iron from the purely metallurgical aspect, it may be regarded fundamentally as the same type of material as normal blackheart malleable iron in the as-cast state, consisting almost entirely of pearlite and cementite, its chemical composition being adjusted to modify its behaviour in annealing. (See Fig. 1). The material is annealed under very similar conditions to those for normal blackheart malleable iron, but, it is in cooling that the difference in composition functions in restraining complete graphitisation and ensures that some of the carbon remains in the combined form to give a finely pearlitic matrix.

Fig. 2 shows a typical pearlitic malleable structure after being subjected to a normal annealing process. To further refine the structure the castings may be subjected to a short additional heat-treatment which considerably refines the structure of the pearlite as shown in Fig. 3.

There is undoubtedly a very wide field of usefulness for this pearlitic malleable, and, although as yet it has not been extensively produced in this country, there is sufficient indication that the demand for it will increase rapidly as soon as its outstanding properties are more fully appreciated.

Among the exhibits of malleable iron castings is one of the largest producers of blackheart malleable castings in this country, Hale & Hale (Tipton), Ltd. Castings for a wide variety of trades and industries are on view together with speciality products for use in coal mining. Particular interest is attached to a new special purpose material known as "Permalite," which is allied to the malleable family and has a tensile strength of 33–35 tons/sq. in., against 20–24 tons for good quality blackheart. This new material is shock-resisting and has good elongation which is indicative of ductility not generally present in high duty cast irons.

These sections include research, production, service problems and choice of materials.

Light Metal Developments

Broadly viewed, a more widespread appreciation of the varied and special utility of aluminium and its alloys has been one of the most compelling forces in its general market expansion since the end of the war. It must be remembered that during the war, the aluminium industry was in a sheltered position as part of a rapidly expanding aircraft industry. During this time its fabricating industry has developed from workshop proportions into a mass producer of castings, extrusions, sheet and forgings. Since 1938 its capacity has been increased five-fold, and, despite many difficulties experienced since the end of the war, output for peacetime applications is increasing rapidly. Indeed, in some fields, such as extrusions and sheet, the capacity of available plant is not equal to the demands made upon it, however, plant developments are in progress which will increase capacity in the near future.

It is noteworthy that aluminium is unique among materials of construction in that its price is lower now than it was before the war. While the prices of other metals have risen the price of primary aluminium has fallen and is now the cheapest of the non-ferrous metals per unit of volume. Apart from this latter fact, the great expansion of the fabricating industry to supply the needs of the aircraft industry has familiarised so

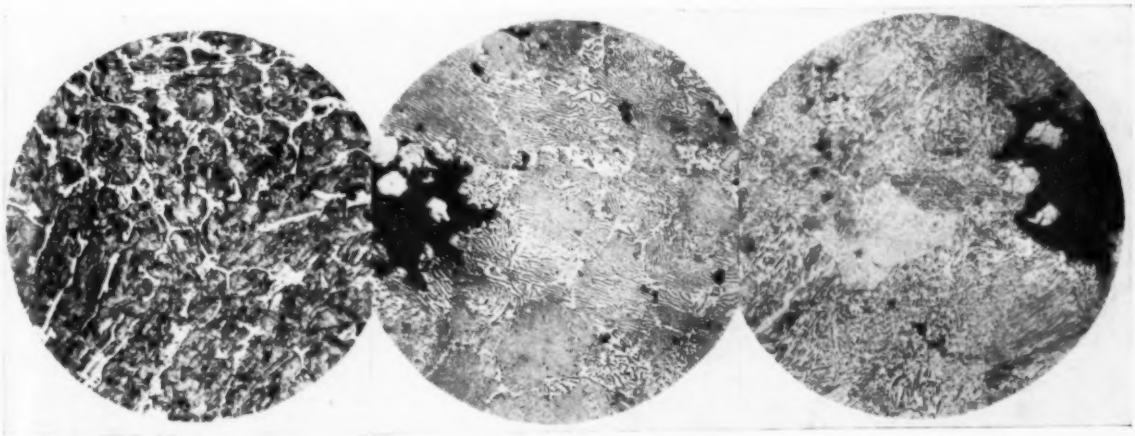


Fig. 1.—Hard white iron before annealing. X250.

Fig. 2.—Showing finely divided pearlitic structure after first operation of double anneal. X250.

Fig. 3.—Finally annealed product showing further refinement of pearlite. X250.

many with the necessary technique, and it is not surprising that the demand for aluminium products of many kinds has been coming from an increasing number and variety of consumers. The aluminium industry realises that only by persistent work can this trend continue, and elsewhere in this issue, Dr. E. G. West reviews some recent developments and discusses some typical applications of aluminium and its alloys.

Although magnesium was isolated several years before aluminium was discovered, relatively little progress was made in its development until about twenty-five years ago; since that time, however, remarkable progress has been made and magnesium-base alloys are being increasingly employed for purposes where their lightness coupled with moderate strength is required. Like the aluminium industry, the magnesium industry has been built on a basis of scientific research and precision control and great progress in production was effected during the recent war. It is noteworthy, for instance, that one large British foundry, specialising in the production of magnesium alloy castings, produced over 20,000 tons of castings during the war, over 16,000 of which were for aero-engine and aeroplane constructors. When it is remembered that magnesium, weight for weight, is four times the bulk of steel, this is no mean achievement.

The expanded usage of the war years has broadened the knowledge and appreciation of the value of the light-weight magnesium alloys in industry. These alloys have outstanding advantages in connection with problems of transport and traction. The power/weight ratio, strength/weight ratio, and pay load are three points which command the attention and occupy the thoughts of most designers and engineers working in the transport industries. But the successful use of these alloys is not confined to the transport industries, they are being increasingly used in the electrical industries, in the machine tool, textile and wireless industries, and by scientific instrument makers. They are also being used for industrial and household equipment, such as air conditioning, conveyers, elevators, excavating equipment, foundry equipment, hoists, furniture, office equipment, photographic and printing equipment and for several other purposes, many of which are on view at this Fair. Particular attention should be given to the exhibits of F. A. Hughes & Co. Ltd.

Nickel and Nickel Alloys

The Mond Nickel Co., Ltd. feature the extensive advisory and research services of their Development and Research Department. The stand is divided into four sections, each illustrating a particular type of service, with actual examples and diagrams, showing typical problems on which advice is available.

A special feature is the Nimonic series of alloys, originated during the war, by The Mond Nickel Co., Ltd., for stressed parts operating at high temperatures in gas turbine engines. These alloys, produced by Henry Wiggin and Co., Ltd., have been a major factor in the lead Britain has achieved in the design of successful jet-propulsion engines. Moreover, they undoubtedly have an important part to play in the development of the gas turbine in other fields, such as marine engineering, land installations and locomotives. Other alloys with special heat-resisting properties, developed for such applications as electric furnace belts, nitriding containers and thermocouple sheaths, are also exhibited.

Tools and Tool Materials

The use of tungsten carbide for single and multi-point tipped tools, milling cutters and wire-drawing dies with the inherent increase of tool life and better finish of product is well known. However, what is not well known is its application for other purposes. The vast number of important applications on view makes it quite obvious that there is no industry in which tungsten carbide cannot be employed for economic increased production over previous materials used. It is also obvious from the claims, substantiated by examples, that tungsten carbide is the answer to the Government's drive for increased production per man and machine hour, at a great reduction in cost and maintenance.

Notable amongst examples of "Wimet," shown by A. C. Wickman, Ltd., is that of interrupted cutting with tungsten carbide. It has long been the opinion of production engineers that tungsten carbide is not economic for this type of machining due to the break-down of the tip because of the shocks imposed. However, a "Wimet" tool is on view that has received to date some 31,000 shocks and is still in good condition. The billet machined with this tool is also on view, together with an unturned billet with machined slots, and the finish obtained is better than that commercially obtained with other cutting tools.

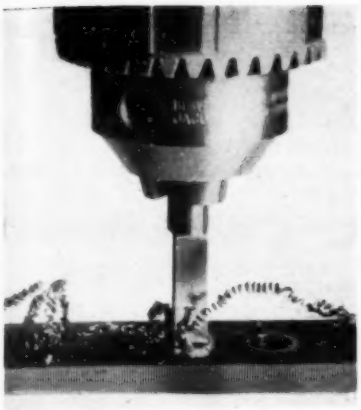
The extraordinary hardness of "Stellite" cutting alloys at red heat is demonstrated by Deloro Stellite, Ltd. This is a cobalt-base alloy which does not soften like steel and to prove this a "Stellite" toolbit heated red hot with an oxy-acetylene torch will be shown turning a steel billet with ease. This property of "Stellite" is used to full advantage in the range of cutting tools and heat-resistant castings and welding rod which are shown. This firm is now supplying a drill which will drill a hardened file to give a clean hole correct to size. The operation only takes a minute or so, and the drill can be used over and over again and reground as required.

Hardsurfacing with "Stellite" is being demonstrated throughout the Fair. A production operator will be at work on a variety of jobs and visitors will be able to see for themselves the ease with which this material can be applied to form a hard surface which is both wear and heat resistant.

A fully representative range of files, machine tools, "Speedient" high-speed drills, reamers, cutters, butt-welded lathe tools, carbide-tipped tools, "Millenicut" files, hack-saw blades, long saws, cross-cut saws, wide and narrow band saws, etc., including the Firth Hardometer hardness testing machine, are a few of the exhibits of Thos. Firth & John Brown, Ltd. A comprehensive range of branded tools is also displayed by Edgar Allen & Co., Ltd., particular attention being directed to tungsten-carbide tipped tools and to butt-welded high-speed steel cutting tools.

Heat-resisting Products

Apart from the special steels developed for heat resisting purposes, many operations involve the use of parts which are required to withstand high temperatures and give good service. For this purpose the Calorised mild steel and Calvert nickel-chrome heat-resisting products of the Calorising Corporation of Great Britain, Ltd., are of particular interest. They comprise heat-treatment containers for cyanide, salt and lead-hardening and annealing purposes. They are also proving valuable



Drilling a file with a Stellite drill.

for nitrate of soda baths for the heat-treatment of aluminium-alloy parts.

A number of specialities in this field are shown by the Wycliffe Foundry Co., Ltd., an associate of Follsain Metals, Ltd., which include sootblowers, carburising and cyaniding pots, pyrometer sheaths, and various furnace parts treated by their Penetral H.T. process, which gives to iron and steel special resistance to oxidation and to sulphurous gases at temperatures up to 1,050° C. Other special heat-resisting parts are also exhibited, as also are castings which possess a very high resistance to wear suitable for the wearing parts of certain types of high-speed pulverising machines.

Melting Furnaces

The Electric Furnace Co., Ltd., were the first in this country to supply arc furnaces, the low-frequency furnaces for non-ferrous melting and high-frequency melting furnaces and the exhibits of this firm are of particular interest in view of practical experience of the war period which is now being applied to post-war developments.

Of considerable interest is the new Birlec-Tama low frequency induction melting furnace for aluminium and light alloys which is being exhibited, for the first time in this country, by Birlec, Ltd. The furnace shown is a relatively small, bale-out type designed for supplying die-casting requirements. The holding capacity is approximately 75 lb. per hour. Larger models are available, however, up to 500 k.w. power input, 6,000 lb. holding capacity and one ton per hour output rate. Although this exhibit marks the introduction of the furnace to the British market, many potential users will be familiar with the success this equipment has achieved in the United States.

In addition to the above furnace Birlec, Ltd. show a small indirect arc-melting rocking furnace. The unit shown is designed primarily for laboratory, development and small-scale production work, and has a charging capacity of 10 lb., but in this instance also large production sized units are available. This furnace is especially useful for melting special alloys for investment casting processes and can attain high temperature for dealing with high-melting point material. The larger production types are widely used for melting alloy iron where close control of analysis and pouring temperature are essential, together with the liberation of occluded gases, as well as for brasses, bronzes, etc.

Pipes and Tubes

The great increase in the number of uses for tubing is one of the most spectacular developments of recent times. By far the largest tonnage of steel seamless tubing is used for carrying gas, oil, water and steam; this must account for hundreds of thousands of tons annually. The increasing use of high pressures and temperatures for steam generation, particularly in land power stations, has led to the growing use of steels for superheater tubes that will stand the severer conditions of service. The choice of material and size is governed by the permissible amount of creep and of oxidation by steam and flue gases. The present tendency is to use the low molybdenum (0·5%) steels with and without about 1% of chromium, in which the arbor is also kept low to facilitate cold-working operations and expanding into tube plates. Sometimes, in heat-interchange work, the stress-carrying capacity is secondary to its resistance to attack by hot gases, and it becomes more important to increase the chromium. More arduous conditions demand the use of austenitic steels possessing to an enhanced degree both creep strength and resistance to scaling such as the 18/8 chromium-nickel family, of suitable composition to resist the disintegration phenomenon known as "weld decay." For the severest requirements even more highly alloyed austenitic steels are available in tube form. In this country the sizes of seamless steel tubes commercially available cover most things between hypodermic needles of a few thousands of an inch up to boiler drums of 36 in. dia. or more.

Copper and copper alloy tubes are displayed by many firms, notably by I.C.I. (Metals), Ltd., who show a comprehensive range of tubes of various sizes and qualities, from copper tubes for domestic water systems to cupro-nickel condenser tubes. Tubes products forms only a part of the exhibits of this firm. The special properties of "Everdur" should be investigated. This brand of copper is particularly advantageous in the manufacture of boilers, cylinders, storage tanks and heaters, and for all purposes connected with water conveyance. It is as strong as steel and can be welded by oxy-acetylene, carbon arc or resistance methods.

A special feature is made of the standard forms in which aluminium and its alloys are produced in the various works of The British Aluminium Co., Ltd., of which tubes and sections play a prominent part. Reynolds Tube Co., Ltd. demonstrate the great advances made in light alloys, not only in the form of tubes, rods, sections, etc., but also in scientific processes of manipulation design to minimise the number of operations. This cutting out of intermediate operations proved of incalculable benefit during the war, not only in time saved but also in the cost. The aluminium alloy "Kynal" shown by I.C.I. (Metals), Ltd., is also exhibited in the form of tube, rod, etc. A comprehensive range of non-ferrous alloys is shown by T. J. Priestman, Ltd., which includes examples of aluminium sand and die-casting alloys, and a range of pre-alloys. Brass and bronzes are also displayed together with gravity- and pressure-cast brasses, high tensile, manganese and aluminium bronzes.

Heat-treatment Equipment

Considerable progress in heat-treament plant and equipment has been made in recent years particularly in the application of induction heating for heat-treatment

A permeable lined heat treatment furnace.

operations. There has been a growing demand for the use of controlled atmospheres in furnaces, but probably the more insistent need has been for furnaces designed to simplify the operation to overcome the difficulty of using unskilled labour. Automatic temperature control is more general to-day than ever, and where the process permits automatic heating, cycle regulators are introduced. Mechanical handling equipment is being increasingly employed to meet the requirements of modern production methods. The greater care in the use of alloying elements in both ferrous and non-ferrous alloys, together with the stringent demands for improved properties, has imposed restrictions on the liberties that can be taken to obtain the desired result, and in many cases improved furnaces have also necessitated improved technique.

Induction heating equipment is exhibited by several firms, and in many cases practical demonstration will be a feature. The highly developed mechanical devices built by Birlec, Ltd., for the automatic feeding of the material to be treated, and the timing of the process are typical of this type of equipment new to the Fair. It includes a small automatic machine for the rapid surface hardening of steel spindles at the rates of about 600 per hour, as well as a larger machine for heating the ends of short bars for upsetting at rates up to 1,000 per hour.

Induction heat-treatment is ideal for mass-production and shows a considerable saving of time and cost. Typical samples of parts treated are shown by Electric Furnace Co., Ltd., and Rediffusion, Ltd., and Philips Lamps, Ltd. This method of locally hardening steel parts was used to a great extent during the war, particularly for swive' pins for caterpillar tank tracks, and this application has been turned to peace-time use in the manufacture of tractors for home and export. High-frequency generators have wide applications for hardening, tempering, annealing, sintering, hard and soft soldering and melting, all of which can be performed with a standard generator.

A permeable lined furnace is demonstrated amongst the gas industry developments at the Fair, which incorporates a permeable refractory lining, the products of combustion being extracted through the refractory by means of a suction applied to the annular space by means of an ejector. The side and back walls, door and arch, are all formed from this refractory. The hearth and supporting piers, and parts of the structure, liable to heavy loading and abrasion, are constructed from best quality firebrick. The whole structure is backed with 3–6 in. of burnt diatomite insulating brick. Under hearth firing is employed by means of six special air blast burners, which ensure that combustion is completed under the hearth and only the products of complete combustion enter the furnace chamber. These products, in passing through the permeable lining, give up some of their heat to it. This demonstration furnace is suitable for all general heat-treatment work at temperatures up to 1,200° C., and furnaces lined with special permeable sillimanite are available for the higher temperature ranges. Many manufacturers are exhibiting some of their products on the British Gas Council's stand showing the application of town's gas in industry. In addition to the permeable lined furnace outstanding gas developments included the application of gas to infra-red drying, and the concentrated combustion burner and brief reference can be made to them here.

Town's Gas in Industry

The infra-red units shown are typical of many which are in use throughout the country for paint drying and similar purposes, and the most remarkable thing about this process is the rapidity with which drying is accomplished. With certain classes of work, it is possible to cut down the drying time from hours to a few minutes, but it should be emphasised that infra-red is not the answer to all drying problems.

The concentrated combustion burner is shown both as a separate unit and also incorporated in a "Cyclone" furnace in which high temperatures are obtainable very rapidly. As the name implies, the chief feature of the burner is that it is capable of burning a large quantity of gas in a very small space, and the resulting high temperature makes it particularly suitable for the local heating of components, such as the faces of hammer heads or jaws of spanners which have to be hardened. When fitted to furnaces it reduces the size of the structure, and furnaces fitted with these burners are used very successfully for general forging, nut and bolt forging, metal melting and other processes.

Metals, Alloys and Chemicals

Products necessary in the manufacture of various grades and qualities of steel are exhibited by Murex, Ltd., including carbon-free ferro tungsten, ferro vanadium, ferro molybdenum, ferro titanium, ferro columbium, tungsten metal, chromium metal, manganese metal, etc., in various sizes and to various analyses. Manganese copper, titanium aluminium, nickel aluminium are some of the wide range of non-ferrous metals and alloys produced, which are also exhibited. "EEL" Brand, a nickel hardened lead base anti-friction metal, which has proved itself suitable for most types of bearings

is shown. Fine chemicals of tungsten, molybdenum and vanadium which are used increasingly in the dye, pigment and chemical industries, are displayed.

The production of powders of high purity and concontrolled grain size for the manufacture of compacts by modern powder metallurgical technique is a growing feature of this Company's activities. Powders used for the manufacture of cemented tungsten carbides are shown, the whole process of reduction from the ore being carried out under careful control in the works. A group of new permanent magnet alloys, generally known as Alnico and Alcomax is also exhibited. The application of powder metallurgy to the manufacture of magnets has overcome a number of difficulties. The principal advantage of the sintering process for making Alnico and Alcomax magnets lies in the fact that shapes, which cannot be cast economically, can often be pressed and sintered. This applies particularly to the small magnets weighing from 1 oz. to fractions of a gram, and magnets with accurate holes for spindles, locating fixtures and the like, and magnet shapes which could only be cast or ground with extreme difficulty.

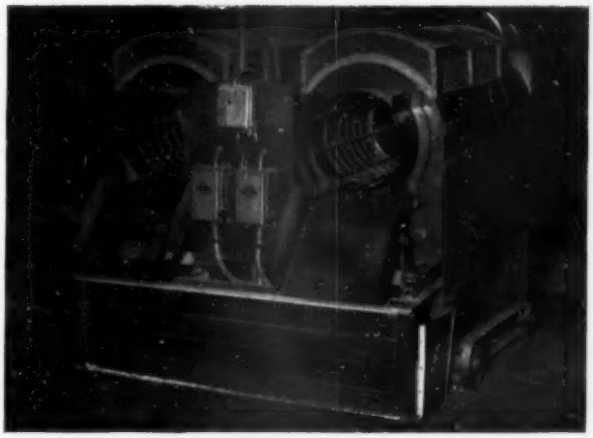
Magnet Machinery

The extent to which the electric magnet is applied in industry is somewhat surprising to the average engineer and yet an examination of the representative range of machinery available for practical demonstration purposes exhibited by Electromagnets, Ltd. will provide convincing evidence that few industries do not use them in one form or another. The exhibits include many improved and new designs of equipment for specific purposes. These may be roughly classified as lifting magnets, as used in steelworks, rolling mills, foundry scrap yards and engineering shops for handling ferrous materials, components and structures; magnetic separators, which are applied in so many industries for extracting iron and steel, either to prevent damage to mechanical handling equipments, feeders, mixers, sifters, crushers, pulverisers, etc., or for purification purposes; magnetic clutches for power transmission; hold-down magnets and chucks for holding ferrous work during machining operations; and magnetic equipment accessories as aids to welding.

Of particular interest is the new "Rotocyl" magnetic separator which Electromagnets are now making under patent licence, and which is suitable for almost universal application. This machine has been developed by practical engineers in the service of one of the largest metal refineries in Britain, who discovered a system of magnetic separation which is a combination of systems, sequenced and blended in such a way as to give the results aimed at. Two of these machines now in operation handle the capacity of seven or eight other machines which have now become redundant, and the saving in labour, space, handling, current and general maintenance is of a very high order.

Testing Machines

Quite a wide range of testing equipment is on view including machines for compression, hardness and impact testing. A range of these machines is exhibited by W. & T. Avery, Ltd., whose range of unusual testing machines is represented by a 50-ton self-indicating machine with hydraulic straining gear, capable of carrying out tension, compression, shear, bend and hardness tests. Simplicity and speed of operation are the keynotes of this machine, which is made in various



Courtesy of Electromagnets Ltd.

The new "Rotocyl" magnetic separator.

capacities. A smaller machine, of 10 tons capacity, is self-contained and requires no foundations other than a strong level floor.

This firm also exhibit a machine for determining the fatigue strength of materials, full-sized machine components, and riveted or welded joints, under three types of loading—viz., pulsating tension or compression of equal or unequal value: pulsating stress imposed on an initial static tensile or compression stress; and pulsating transverse bending imposed on an initial static bending stress. The machine has a capacity of 20 tons.

A new 25-ton universal testing machine by Tangyes, Ltd., has the following outstanding features: it will permit all types of tensile, compression, beam, shear and bending tests; enable many precision pressing operations, such as bushing, forming, straightening, etc., to be carried out; incorporates a new type of precision load indicator; is entirely self-contained and requires no foundations; is extremely simple to operate and service; and provides full protection to the operator.

The load measuring device, used in the machine, is entirely separate from the hydraulic straining system and is capable of measuring the applied load with very great accuracy. It is of the metal diaphragm type, hydraulic pressure being generated behind the diaphragm without internal friction, resulting in an accurate load indication. The whole unit is hermetically sealed and once fitted in the machine requires no attention. Accuracy is further ensured by providing special self-aligning mountings for the tensile and compression testing members, thus ensuring that the specimens are at all times truly loaded. Tangyes are demonstrating this new machine at their Cornwall Works, Smethwick, Birmingham, so that those interested can see the machine in operation.

Gears and Gear Units

Gears and gear units play such an important part in every industry that their manufacture has for long been regarded as a highly specialised industry. Representative of this field of activity is David Brown & Sons (Huddersfield), Ltd., who, with their associate firms, exhibit complete gear units comprising members of the Radicon worm-reducer family, single reduction helical gear units, Roloid pumps, a marine reverse gearbox,

Machine for determining fatigue strength of materials.

automobile gearboxes, steering units, worm gear and spiral bevel differential units, while the separate loose gears include spur, spiral, straight and spiral bevel, single and double helical gears, industrial and automobile worm gears, automobile spiral bevel and hypoid gears and differential bevel gears.

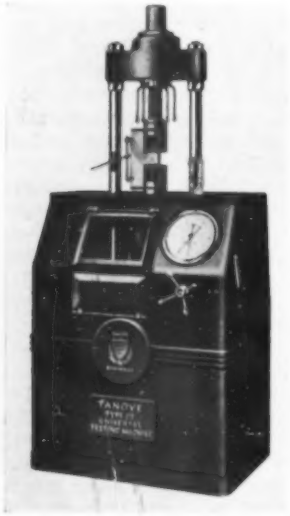
Of the many David Brown transmission media and ancillary equipment, two instruments merit special mention because of the assistance they can give to the British engineering industry in keeping ahead of its competitors. Carefully co-ordinated research in the laboratories of this gear manufacturing organisation has produced the Topograph and applied, by means of the Polariscope, the principles of the photo-elastic method of stress analysis to gear tooth design. Use of the Polariscope facilitates accurate determination of the maximum stresses to which materials are subjected in service. The Topograph has been developed as an accurate and economical means of recording the roughness of any suitable material surface. Operating on a simple pneumatic principle, it gives a pen record of the profile of the surface along a selected straight track and is easily adjustable to suit any class of surface normally used in modern engineering practice. Irregularities of the order of 2 micro in. are readily discernible when the magnification of 20,000 to 1 is used.

Welding Equipment

The extended use of welding processes, using gas or electricity, is helping many engineering firms to increase output, simplify assembly operations and to effect economies in time and material. The immense importance of welding during the war was recognised by all the leading engineering authorities and to assist users and potential users of welding for peace-time products information services are available from the welding sections of the British Electrical Allied Manufacturers Association, or from the British Oxygen Co., Ltd. Visitors to the Birmingham section of the Fair will not only see many examples of welding, but also many types of welding equipment, and will be advised on the use of various welding processes by technical representatives of the many firms exhibiting.

Typical of the exhibitors in the electric welding field is Murex Welding Processes, Ltd., who show a wide range of electric arc welding plant, accessories and electrodes. A mobile 400 amp. diesel engine driven set as supplied in large numbers for work now extensively carried out in oilfields. Special precautions have been taken in the manufacture of this plant to ensure that it functions satisfactorily in the extremes of ambient temperature and altitude under which they have to operate.

The exhibits include a 250 amp. self-contained welding transformer, and a 300 amp. motor generator set complete on a trolley and fitted with a remote control regulator. In addition a full range of welders' accessories is on view, and electrodes displayed include those used for welding mild steel, high tensile steels and heat-resisting steels. non-ferrous metals, cast iron and special electrodes for hard-facing applications. Demonstrations are given in the welding of ferrous and non-ferrous metals, and visitors who have welding problems should take this opportunity of discussing them with experts.



New 25-ton universal testing machine by Tangyes.

Courtesy of Murex Welding Processes Ltd.

A 400 amp. diesel engine driven electric arc plant.

Developments in the Applications of Controlled Atmospheres

By I. Jenkins, M.Sc., A.I.M.

(Research Laboratories of The General Electric Co., Ltd.)

The application of controlled atmospheres in heat-treatment operations has increased considerably during the war years, and the added experience gained has confirmed their industrial importance. Initially developed in connection with bright annealing the field of application now includes almost every heat-treating process, and this review of achievements in this field indicates how much reliance is now placed upon the control of atmospheres in such operations and the trend of further developments in their applications.

Introduction

CONTROLLED atmospheres are now well established in the heat-treatment field, and during the past twenty years have been so widely publicised that there should be few, if any, associated with heat-treating processes who have not become familiar with the subject. The various types of atmospheres and the corresponding generating equipment have been described fully and repeatedly, together with the chemical reactions on which their applications are based and the attendant equilibrium data on gas metal systems.

The time when a controlled atmosphere process was considered as being synonymous with "bright annealing" has passed long since, the field of application having extended so considerably beyond this initial phase that it now includes almost every heat-treating process in which reactions between gases and metals call for some measure of control. Much impetus and added experience were inevitably gained during the war years when the demand for heat-treatment plant with associated controlled atmosphere equipment was considerable and when the general success of such installations confirmed the increasing industrial importance of the subject.

Perhaps the present time, therefore, is opportune to review briefly what has already been achieved and to consider what further development can be anticipated in this field.

Present Practice

(*a*) *Atmospheres.*—The various types of industrial controlled atmospheres are now fairly well standardised (Table I), and in this country include those derived from synthetic ammonia and from the controlled combustion of a hydrocarbon gas such as town's gas, propane or butane. The use of charcoal as a source of a controlled atmosphere has not developed to the same degree, mainly because of the greater supervision necessary and difficulties in stabilising the composition of the generated gas, especially when an atmosphere is required with a low concentration of decarburants. The use of charcoal as a means of processing atmospheres prior to use for carburising or the heat-treatment of carbon steels has been maintained, however, and will probably extend.

Controlled atmosphere generators, as at present designed, are compact, self-contained units and generally speaking are automatic in that once set to deliver an atmosphere of the required composition, they continue to do so with the minimum of attention other than periodic inspection and routine maintenance. The use of automatic gas analysis instruments is increasing, and is to be encouraged, being complementary to the less frequent complete chemical analysis of the generated atmosphere. A continuous indication of one constituent of the atmosphere—usually CO_2 or H_2–, when related to the detailed analysis, is usually sufficient to maintain the correct working conditions and ensures a more positive control over the process as a whole.

(*b*) *Furnaces.*—A controlled atmosphere process is essentially one which ensures a measure of control over the composition of the atmosphere inside the heating chamber and, thereby, over the reactions between the metal and the gases surrounding it. This is most readily ensured by producing, external to the furnace and under closely controlled conditions, an atmosphere which is then fed into the heating chamber to replace completely the natural gases which would otherwise be present. In this respect it is obvious that furnace design is a primary consideration, since not only must there be facilities for the introduction, circulation and discharge of the controlled atmosphere, but above all the atmosphere must not be contaminated by gases entering the furnace chamber from other sources.

Industrial controlled atmospheres—not, perhaps, as we know them to-day—were available many years before furnaces were deveolped in which they could be successfully and economically used. This raises an important point which cannot be stressed too heavily—that the development of a controlled atmosphere process is closely allied with that of the modern heat-treating furnace, and only when used in association with the latter can the maximum advantages of the technique be achieved. With some ingenuity and a modification in furnace design, modern controlled atmospheres can and are being used on older-type installations, but such can only be an unsatisfactory compromise and in the long-term view, uneconomical.

The early industrial development of the controlled atmosphere process was associated with the introduction into industry about 25 years ago, of the electric resistance furnace for general heat-treatment work. Since that time, developments in the design of furnaces, heated by electricity or fuel-fired, have been very rapid, and at the present time there is a wide variety of furnaces available to industry, many of them of a general-purpose type in that they are suitable for a number of applications, others designed for a specific purpose. It is not possible, in an article of this kind, to discuss the various types in any detail, but certain generalisations can be made

The main requirements of a furnace from the point of view of a controlled atmosphere are gas-tightness and adequate distribution of the atmosphere inside the furnace chamber. The manner in which the latter is achieved depends to a large extent upon the nature of the work being treated. Where this is solid and bulky, atmosphere circulation induced by natural convection is usually adequate ; where the charge is a collection of small articles representing a high ratio of surface area : weight, forced air circulation as by a high-speed fan may be necessary, having the further advantage of improving both the rate of heating and temperature uniformity.

Modern furnaces are compact units, fitted with automatic temperature-control gear and instruments which indicate or record both the furnace temperature and that of the work being treated. Complex heating and cooling cycles can be catered for with program controllers. Cleanliness and simplicity of operation are features which automatically lead to greater efficiency in the process as a whole, whilst the maximum rate of heat transfer to the work, uniformity of heat-treatment and low overall running and maintenance charges are the results of careful furnace design and development.

(*c*) *Applications.*—The fields of application of controlled atmospheres can be conveniently grouped under the following headings :—

1. Replacement of old processes.
   (*a*) Processes which require the maintenance of the original surface finish and of the concentrations of surface constituents.
   (*b*) Processes which require the chemical modification of the surface.
2. Development of novel processes.

Group 1 (*a*).—Within this group are included processes for the bright annealing of ferrous and non-ferrous metals and alloys, and the bright or clean hardening and tempering of carbon and alloy steels with freedom from decarburisation, either as re-rolled products or in fabricated form. Details of the various atmospheres recommended for each particular application are given in Table I, and the techniques are now so well established that they call for little further comment here, excepting for one or two specific aspects.

Thus, for example, whilst heat-treatment of carbon and alloy steels, with freedom from decarburisation, can now be achieved quite readily in a suitable controlled atmosphere, it is unfortunately, frequently the case that the material is already in the decarburised condition prior to heat-treatment. It is possible, however, by suitable selection and control of the furnace atmosphere to promote re-carburisation of the steel, up to the original carbon content during the final heat-treating process, thus permitting of the full development of the physical and mechanical properties of the material on

TABLE I.—Industrial Controlled Atmospheres

Atmosphere	Air : gas ratio	Approximate composition (volume %)						Cost per 1,000 cubic feet	Characteristics	Main applications
		CO_2	CO	H_2	CH_4	N_2	Dew point			
Dissociated ammonia.	—	—	—	75	—	25	−50° C.	15s. to 20s.	Strongly reducing and explosive with air.	Bright annealing stainless alloys and high carbon steels. Clean annealing brass.
Burnt ammonia.	2·5 : 1	—	—	20	—	80	Room° temp. to −50° C.	7s. 6d. to 10s.	Reducing. Just combustible.	Bright annealing carbon steels and non-ferrous metals. Clean annealing brass. Bright hardening on short time heating cycles.
Nitrogen from ammonia	3·75 : 1	—	—	—	—	100	Room° temp. to −50° C.	7s. 6d. to 10s.	Inert and non-combustible.	Bright annealing and bright and clean hardening carbon steels. Annealing silicon steel.
Reacted gas.	1·25 : 1† (For town's gas)	0·0	24·5	35·5	1·5	38·5	−25° C.	3s. 6d. to 4s. 6d.	Strongly reducing, toxic and combustible.	Bright and clean hardening. Brazing carbon and alloy steels. Carburising.
Partially burnt fuel gas.	2 : 1†	4·5	9·8	15·0	0·2	70·5	Room° temp. to −50° C.	1s. to 1s. 6d.	Reducing. Just combustible and toxic.	Bright annealing mild steel. Bright brazing and soldering mild steel.
Completely burnt fuel gas.	4·5 : 1†	10·0	0·5–1·0	0·5–1·0	0·0	87–89	Room° temp. to −50° C.	1s.	Slightly reducing. Non-combustible.	Bright annealing copper and high copper brasses. Copper brazing.
Completely burnt fuel gas, scrubbed of CO_2 and H_2O.	4·5 : 1†	0·0	0·5–1·0	0·5–1·0	0·0	98–99	−50° C.	2s. 6d.	Slightly reducing. Non-combustible.	Bright annealing carbon steels and low alloy steels. Bright and clean hardening carbon steels.
Completely burnt fuel gas, treated over hot charcoal.	4·5 : 1†	0·0	20·0	2·0	0·0	78·0	Less than −20° C.	1s. 6d. to 2s.	Just combustible. Reducing and toxic.	Bright annealing high carbon steels. Bright and clean hardening carbon steels. Diluent for carburising atmospheres.
Charcoal gas. Low temperature auxiliary heating.	—	20·0	—	—	—	80·0	Room° temp. to −50° C.	1s. 6d.	Non-combustible.	Bright annealing copper. Copper melting.
Charcoal gas. High temperature auxiliary heating.	—	—	34·0	—	—	66·0	Less than° 20° C.	2s. 6d.	Reducing combustible and toxic.	Bright annealing carbon steels. Bright and clean hardening carbon steels. Diluent for carburising atmospheres.

° Dew point of wet gases may be red need if required from room temperature to less than −50° C. by means of suitable drying agents.
† Determined by the composition of the fuel gas.

subsequent hardening, without the necessity of removing the decarburised surface zone. The surface carbon content can be controlled within close limits by the use of sensitive instruments which indicate the so-called "carbon potential" of the furnace atmosphere—i.e., the carbon content, at any temperature, with which the atmosphere is in equilibrium. In view of recent work on the adverse effect of decarburisation on fatigue resistance of steels, the carbon restoration process is a particularly important development.

The bright or clean hardening of carbon steels is now carried out in furnaces so designed that the work is surrounded by the controlled atmosphere up to the moment when it enters the quenching medium. The order of surface finish obtained is largely related to the size of the work being quenched. Where this is small and the resulting quenching rate very rapid, bright hardening is possible; massive parts, however, because of their slower overall cooling rate are susceptible to staining due to reaction with the quenching medium. Alloy steels, including the martensitic stainless type, can be bright-hardened in continuous furnaces by quenching in a jet of a suitable controlled atmosphere, which, if need be, is refrigerated in order to ensure a high quenching rate.

The patenting of carbon steel wire has also been brought within the field of application of controlled atmospheres, the wire being protected against oxidation and decarburisation up to the moment of quenching in the lead bath. However, developments in the use of direct heating of the wire by using it as an electrical resistance might eliminate the necessity for a controlled atmosphere. High alloy steels, including the stainless variety are most conveniently treated in continuous furnaces, the protective atmosphere invariably being cracked ammonia. Specially designed furnaces, such as the catenary type, ensure that the material does not come into contact with any internal part of the furnace, this ensuring that the high degree of surface finish is maintained on the final product.

In the non-ferrous field, copper, nickel and their alloys, as well as the red brasses and alloys containing less than approximately 10% of zinc are bright and fully annealed without difficulty, in both burnt fuel gas and the ammonia type of atmosphere. Developments in desulphurisation processes and fundamental investigations on such problems as the hydrogen-embrittlement of copper are but two important factors which have assisted considerably in firmly establishing these processes on an industrial scale.

Group 1 (*b*).—The two main processes covered by this group are the carburisation of steel and the malleablisation of white cast iron. Pack carburising and malleablising in iron ore have one feature in common—namely, that at temperature the mechanism of both processes is essentially gaseous. The packing material, with occluded air, is the source of the initial atmosphere in the heat-treating container, and therefore it serves merely as a reservoir to supply, to the metal surface via the gas phase, the reacting element—i.e., carbon in carburising, and oxygen in malleablising.

It is obvious, therefore, that both these processes lend themselves fundamentally, to the application of a controlled atmosphere technique. Developments in gas carburising have been proceeding over a number of years, both from the fundamental and from the practical points of view, and much information is now available to ensure a successful industrial application. A number of processes have been developed, differing only in the method of generation and the constitution of the carburising medium. Coincidental with the work on essentially gaseous media, developments have also taken place in the use of liquid carburising agents which are vapourised on entry into the furnace chamber and potentially, are very strong carburisers. All the processes at present available result in a very high rate of carburising with sufficient flexibility to control both the final surface carbon concentration and depth of case. Furnace design ensures adequate circulation of the gases around the charge leading to uniformity in carburisation and clean, soot-free work; facilities are usually provided for the removal of test samples direct from the furnace chamber, so that the progress of the treatment may be followed.

The gaseous malleablising process was developed during the war and is unique in that the present practice does not make use of a separately produced controlled atmosphere. On the other hand, the charge of iron, being rich in carbon, is permitted to produce the initial atmosphere by reaction with the air originally present in the furnace chamber. The resulting carbon monoxide is then regenerated by the addition of an oxidising agent, such as air or steam, the degree of regeneration being controlled so as not to produce conditions which will lead to oxidation of the iron.

Both the gaseous carburising and malleablising processes result in a considerable reduction in times of treatment to effect a given result as compared with the older pack processes, whilst leading to greater efficiency, a more consistent and cleaner product, and greater flexibility and control in operation.

Group 2.—Perhaps the most well-known process within this group is nitriding, which has been a recognised industrial technique for very many years for the production of hard, wear- and corrosion-resisting surfaces. It was probably never considered that nitriding would supplant carburisation; the two processes are complementary, each having its own field of application with a certain degree of overlapping. Little further comment need be made here, other than to say that fundamentally, the process has changed but little since its inception. although much subsequent data has been made available on the mechanism of nitride-hardening, without providing the complete answer to the problem.

Also within this group can be included the gaseous deoxidation of hot-rolled metal strip. This has been confined, in the main, to treatment of hot-rolled low carbon steel strip in continuous, roller-hearth furnaces, the deoxidation, usually in burnt ammonia, being combined with a normalising treatment. Complete reduction of the scale is readily achieved, resulting in a clean, matte-finished surface.

More recently, the I.C.I., Ltd., have developed a sodium hydride process for descaling and de-enamelling. The descaling process is a combination of a salt-bath treatment (sodium hydroxide) and a controlled atmosphere (cracked ammonia), and has proved to be very efficient for the descaling of stainless steels.

Controlled atmospheres are also finding considerable application in the sintering of metal powder compacts, in both batch and continuous mesh-belt furnaces, and, usually as cracked ammonia, in the heat-treatment of sundry valve parts in the radio industry.

Economies

The use of a controlled atmosphere in conjunction with a modern heat-treating furnace has many attractive features when compared with the older methods. Certain of the economic advantages are, perhaps, obvious and may be listed as follows :—

1. The elimination of a subsequent cleaning process, with its unattractive working conditions and fairly high labour and maintenance charges.
2. A saving in material which, by older methods of heat-treatment has to be removed by a cleaning process and which can be recovered only with some effort and additional cost. This saving in material can be quite appreciable—for example, on one particular plant annealing steel castings, the loss due to scaling in furnace flue gases exceeded 5% by weight of the charge.
3. The elimination of heavy heat-treating containers or boxes and of solid packing materials, resulting in improved heat transfer, a saving in fuel consumption and a much quicker turn-round of material.
4. A saving in floor space and improved working conditions which are the natural outcome of the elimination of cleaning processes and of solid packing agents.

Apart from the above, however, there are other advantages, perhaps not quite so obvious, but none the less important. Thus, for example, maintenance costs on modern furnaces and atmosphere generating units are very small, this being particularly so on electric-resistance furnaces, since, per ton of work treated, the cost of the replacement of resistors used in air is insignificant, in view of their long life.

The accurate measurement of charge temperature, together with automatic temperature control devices gives considerable flexibility and does permit, where necessary, of controlled heating or cooling cycles. Labour charges are very much less than with older installations—for example, a batch-type installation annealing 1,000 tons of steel strip, in various coil sizes, per week and comprising sixteen furnaces is handled by three shifts daily, each of three men, one of whom is a crane driver. This labour is adequate for the loading and unloading of annealing containers, as well as for the various operations involved in the actual annealing.

The quality of the heat-treated product is much superior than that produced hitherto. A bright annealing process, for example, retains the original highly-polished surface of cold-rolled strip, whilst the consistency which is obtained obviously results in a great saving in overall cost, since there are virtually no rejects on the grounds of non-uniformity of surface finish.

Future Developments

Whilst the present application of controlled atmospheres is very wide, there is every indication that it will extend still further, both in replacing old equipment and methods, and in the development of entirely new processes. Furthermore, during the past twenty years the increasing industrial importance of the subject has greatly stimulated research on related problems, not only in connection with the generation and application of atmospheres, but also on the rather more fundamental study of gas-metal systems. Within the present-day industrial controlled atmosphere field there remain many problems of practical, as well as academic interest awaiting further study, and these will naturally increase as further practical applications develop.

(*a*) *Atmospheres.*—There is scope for further fundamental research on the physical chemistry of the generation and purification of controlled atmospheres, especially in connection with the development of atmospheres to meet specific requirements, as, for example, carburising and the heat-treatment of carbon and alloy steels.

Simplicity of plant and of operation should be the ultimate aim of all controlled atmosphere work, and the streamlining of the design of the generating equipment can be anticipated. It is also probable that with developments in metallurgical research, even closer control of the atmosphere composition may be required for specific purposes. For example, to maintain a surface carbon content to within $\pm$ 0·025%, which is not an unusual present-day requirement, might involve controlling the concentration of certain constituents of an atmosphere to within $\pm$ 0·25% or less. Here there is room for the development of atmosphere control instruments, and eventually the acceptable design for all atmosphere units might be that in which the operation of the generator is synchronised with a gas analysis indicator so that there is automatic correction for any deviations from the required gas composition.

(*b*) *Furnaces.*—In spite of the wide variety of controlled atmosphere furnaces now available, there is much scope for further development, and indeed, progress in the next few years will probably be to a very large extent, associated with furnace design. Problems associated with temperature distribution within the charge—the desired limits of which are ever decreasing—and gas circulation, are now more often than not specific to the particular application. This is due to the variety in shape and size of the article to be treated, as well as to the more precise heat-treatment schedules which have to be followed in order to develop fully and uniformly the desired structural characteristics and mechanical properties in the material being treated. Already there is practical evidence that such demands are not outside the ingenuity of the furnace designer, without detriment to the economic aspects, and further interesting and original developments in furnace design can be anticipated.

Heat-resisting materials form an important part of modern furnace construction, and there is much scope for fundamental study on the effect of the various industrial controlled atmospheres on the high temperature strength and corrosion resistance of such materials. High temperature strength is the main requirement in that the higher this is, the lower the initial cost of the installation and the lower the running cost because of the lower quantity of extraneous material involved. In addition, the longer life obtained also reduces the overall running costs. The data at present available on corrosion resistance, usually relates to the performance of an alloy in air, and in many cases, this is not relevant to the particular application. A wider knowledge of the performance of heat-resisting materials in controlled atmospheres would be of great practical value, and would probably suggest developments of new alloys and increase still further the service life obtained.

(*c*) *Applications.*—Within the field of bright annealing of both ferrous and non-ferrous metals there are many problems on which further investigation is necessary. Thus, for example, there are indications that the mechanical properties of low carbon steel can be influenced appreciably by the composition of the bright-annealing atmosphere, and it is within the bounds of

possibility that the very optimum conditions of treatment have yet to be definitely established.

Experimental and practical results have demonstrated that a bright annealed surface, especially on ferrous alloys, is unsuitable for subsequent coating, as by tinning or galvanising. The nature of any inhibiting film is not definitely established; it has been vaguely described as being an oxide. A fundamental study of this inhibiting effect might well point the way to a practical solution without having to resort to a light pickling process prior to coating.

The full and bright annealing of yellow brass on an industrial scale has yet to become an accomplished fact. Many techniques have been developed on a laboratory scale, but the translation of the processes to a production basis has met with little or no success. The problem has been explored so thoroughly that it is difficult to foresee any further novel process which will overcome all the difficulties inherent in the problem; the practical solution might well rest with the furnace designer in developing a suitable furnace in which one of the laboratory techniques can be successfully applied.

A problem common to almost all bright annealing processes is the adverse effect of rolling and drawing lubricants upon the degree of surface finish obtained, especially in batch furnaces. Premature cracking of vapourised lubricants before they are swept out of the container leads to sooting, alkali residues, and sulphur-staining of non-ferrous metals. A certain amount of fundamental work has already been carried out and techniques have been developed for minimising the effect, as for example, a slow initial rate of heating, a high rate of gas purge, and the coating of the interior of containers with metals which do not catalyse the breakdown of the oil vapours. In spite of this, lubricants are always potential sources of staining. Degreasing is a positive, but often an expensive solution, and the problem may profitably be followed-up, initially by correlating the lubricating properties of the conventional oils used with their resulting effect upon bright annealing.

A problem, among others in the field of alloy steels, is that of the annealing of silicon-iron or transformer steel. Whilst modern furnaces are particularly suitable for the controlled slow cooling rate required after annealing, the problem of the uniform reduction of the carbon content of the iron, which is already low, to a negligible value, in order to improve the magnetic characteristics, is a very real one. The difficulty would appear to be associated both with the protective nature of the surface films on the iron, and that of ensuring uniform treatment over the surfaces of the sheets.

Both gaseous carburising and malleablising are, in this country at least, comparatively new developments, and within each process there is a large field for further work on both a practical and a more fundamental scale. This is particularly so in gaseous malleablising where, as in other fields, the greater flexibility and control obtained has stimulated research into many fundamentals inherent in the metallurgy of the process, and which have lain dormant because of the lack of a practical method in which the results of any fundamental work could be successfully applied. Thus the foundryman can expect useful data in the near future on the effects of heating and cooling rates and of gas content on the mechanical properties of whiteheart malleable; of the optimum annealing temperature for maximum efficiency; and on the mechanism of graphitisation and the factors affecting decarburisation of the iron.

In practical gas carburising, further developments can be anticipated in the nature of the carburising media used, and in simplification of plant and control. The acceptance of the process on an industrial scale might well prompt the steel manufacturers to further their investigations into the development of steels of controlled grain size, since the new process is ideally suited to the direct quenching of the work from the carburising temperature. Much has already been learned of the mechanism of carburising and of the gas-metal surface reactions involved. Much remains for continued investigation, both with regard to the surface chemistry of the process and the diffusivity characteristics of carbon in plain and in alloy steels.

A field which, from the point of view of controlled atmosphere application, has barely been touched on, is that of the production of protective oxide films on alloys, by the selective oxidation of a readily oxidisable constituent. The contribution of such films to the surface stability of alloys for high temperature service is of fundamental importance to the modern engineer, and obviously practical and controllable methods for producing such films come within the scope of a controlled atmosphere process. Within the same category can be included, for example, the impregnation of metal surfaces with other metals deposited from the vapour phase at elevated temperatures, with a view to improving wear and corrosion resistance as well as general surface stability. In both of these fields, the scope for fundamental research and development work is considerable, the latter involving the furnace designer as much as the specialist in the physical chemistry of the processes involved.

The above are but a few of the many lines of research and development work that lie ahead in the controlled atmosphere field. Many such investigations are well advanced, and already show signs of leading to interesting and novel developments.

New Power Stations

In connection with the programme of expansion of electricity generating capacity in Great Britain, Dorman Long & Co., Ltd., are supplying the structural steelwork and other materials for three new power stations, and for extensions to thirteen existing power stations. Many of these extensions are equal to or larger than the existing units and will mean an increase of double or treble the existing capacity.

Approximately 35,000 tons of structural steelwork alone is involved and the total value of these contracts is about £2,000,000.

The new power stations are Meaford (Stoke-on-Trent), Staythorpe (Newark) and Birkenhead. Extensions to power stations which Dorman Long have on hand include Nottingham, West Ham, Huddersfield, Ferrybridge, Bradford, Stourport, Littlebrook (Kent), Fulham, Thornhill (Dewsbury), Hayle, Earley, Rotherham, Dunston.

Lead Hardening

By Bernard Thomas, F.Inst.P., F.Inst.F., A.I.M., A.I.Mech.E.

To avoid the effect of air or furnace atmospheres in the heat-treatment of ferrous tools and components, the use of fused baths is quite common, and molten lead is frequently used for this purpose since temperatures up to 820° C. can be operated successfully. Clean and pure lead does not alloy or attack steel and, as no "atmosphere" is present, there is neither scaling nor decarburisation of the component within the bath. In this article are discussed the technique of lead hardening, the life of lead pots, brief reference to quenching mediums and to the use of the salt bath for tempering.

LEAD hardening, as a title, rather tends to suggest a non-ferrous subject, but actually covers a field of ferrous activity which receives all too little attention having regard to its interesting nature.

Probably originating in Sheffield where it appears to have first been employed for the hardening of files, the process has spread to other industries where the hardening of small tools is required particularly of selected areas. Amongst such are included: Picks, forks, shovels, trowels, chisels, augers, axes, wedges agricultural and plantation hoes. With all such named tools it is essential that individual attention shall be given to each in quenching to avoid the risk of soft patches which are all too frequent in non-alloy steels when hardening in batches, often by charging machines, such as is done in the case of drop-forgings and stampings.

The batch type furnace (or muffle as often erroneously called) is not satisfactory for individual withdrawal, partly because of the great heat loss when the door is opened, which chills the remaining tools, and partly because of being too slow in heating up. In addition there is the difficulty of door manipulation. A lead-hardening furnace meets these points admirably and allows of a skilled man hardening 1,500/1,600 tools per shift, each receiving individual attention and thus attaining maximum consistency.

A furnace suitable for the work consists usually of a rectangular vessel, or pot, approximately 24 in. in length × 14 in. wide × 16 in. deep, having a wall thickness of $\frac{5}{8}$–$\frac{11}{16}$ in., in which is contained the lead. Heated by gas or electricity (coal and coke was used twenty years ago) the pot is encased either in a bricked surround or sheet metal structure with external dimension of roughly 6 ft. long × 4 ft. wide × 2 ft. 6 in. high. Tools are dipped in the molten lead, submerged to the required extent and held in place by means of a rack until thoroughly soaked, whereupon they are withdrawn and quenched. A hood is necessary over the top of the pot to remove fumes (if any) from the lead and also chimneys to exhaust waste gases where gas heating is employed.

Lead Pots

Cast iron was first employed for the manufacture of the vessels in which to hold the lead for hardening, but quite apart from the immediate failures experienced through presence of blow-holes or porosity, the "life" was complete with monotonous regularity every 15/20 working days. With the casting weighing around $3\frac{1}{2}$ cwt. ($\frac{3}{4}$–$\frac{7}{8}$-in. walls used for cast iron) the cost was not excessive for actual replacement, *but*, what was not so obvious at first sight was that something like 15 cwt. of molten lead would percolate throughout the jointing of the brickwork, and anything from one-third to one-half was lost. Beyond all this was the inevitable loss of production of at least two days—one to cool down and one to rebuild—which is now an important factor.

Experiments of wide nature have been carried out in order to establish the most suitable material for pot manufacture, and from these emerged the fact that the holding of molten lead at a suitable hardening temperature is one of the most exacting duties possible. The molten metal appears capable of finding its way through walls of a casting which have withstood hydraulic tests up to 100 lb./sq. in. Alloys of nickel and chromium offer the longest average life, and in the following table is given the composition of an alloy which may average 12/18 months of service.

	%
Carbon	0·25/0·35
Silicon	2·00/2·50
Manganese	0·80/1·00
Nickel	64·0/66·0
Chromium	18·0/20·0
Tungsten	2·0/ 2·5
Iron	Balance

Table 1.—Composition of nickel-chrome alloy for lead pots.

An interesting point in connection with the composition given is that initially tungsten was not present and trouble was often experienced through the lead percolating slowly through the base of vessels when at work. The structure of the alloy appeared lacking in density and the addition of tungsten to the extent of 2·0% remedied the matter very successfully.

Lower alloy content metals were not very successful, but it is a curious fact that pressed mild steel pots, reinforced around their top edges with a 4-in. flange, and calorised, can always be depended upon to last 20/26 weeks without fail, whereas the highest alloy casting of potential longest life is always likely to fail without reason within a few hours of being put to service.

With any type vessel, however, if success is to be obtained reasonable care should be exercised in re-heating at shift commencements, particularly after a week-end, otherwise cracking may occur. It is advisable, therefore, to spend about 2 hours each morning to re-heat from the approximate 100° C. to working temperature and at least 3 hours after a week-end.

Working temperatures of the lead will, of course, vary according to work being handled and composition of steel involved, but range usually from 770°–820° C.

Molten lead will "boil" at approximately 845° C. which is not only potentially injurious to the operator through risk of lead fumes, but wastes the lead through oxidation. Actually the "boiling" appearance is due to ebullition of gases since the true boiling point is much higher.

Lead Oxide

The surface of the molten lead in the vessel will oxidise at any working temperature if left unprotected, but by covering to a depth of 1–1½ in. with some carbonaceous compound such as granulated wood, or animal charcoal, or ⅜ in. to dust size anthracite (preferred) any oxides formed are reduced back to metal. Usually it is the reddish-yellow amorphous type Pb_2O_3 which occurs, since the higher oxide, Pb_3O_4 is susceptible to decomposition above 470° C. The reaction between the oxide and the carbon is as under :—

$$2Pb_2O_3 + 3C = 4Pb + 3CO_2$$

A precaution of the above nature will maintain a surprisingly clean surface on the bath.

As regards choice of lead to use, virgin pig, although higher in price is invariably better than scrap sheet or tubes, since the latter not only seems higher in impurities, but having been "weathered," forms a scum on the surface which clings tenaciously to the steel surface being treated and will either look "messy" if not dealt with, or quickly ruin the grinding surface of wheels, mops or bobs if such treatment is required after hardening. For this reason, in any case, cleaning of the bath surface daily is an important necessity.

Preparation of Work

Best results are to be obtained by grinding, or scurfing, the steel surfaces prior to immersion in the lead bath, since scaled surfaces such as produced by forging, annealing, re-heating, etc., not only cause the lead to adhere to a surprising extent, with the attendant loss of "carry-over," but worse still, such adherence causes a "blind-spot" on quenching, preventing effective hardness penetration and naturally gives rise to soft spots.

Even on the scale-free surface lead may adhere to a lesser extent, but on such surfaces a preventative may be used in the form of lamp black suspended in kerosene. In this connection not all lamp-blacks are suitable since some will not remain in suspension more than a few seconds. Of the right type, 2½ oz. per gallon will be found to be ample.

Soaking Time and Limitations

In bath-type furnaces one normally thinks of soaking times to the order of an hour per inch, plus the time for bringing up to temperature. With the lead pot, however, there is no comparison, since sufficient tools are immersed in the lead to ensure that each has a total immersion time of not more than four to five minutes and, subject to all other considerations being in order, this is sufficient to obtain a fully martensitic state on quenching. Few if any, other types of furnace can claim this efficiency.

Limitations exist in regard to applicability of the lead pot furnaces. Plates of approximately 0·120 in. thickness are about the maximum that can be hardened thoroughly. Beyond this thickness a tendency for a pearlitic core develops, unless, of course, the pieces are quite narrow in width, as a file, for example, or perhaps high in carbon, or even alloy steel. In the experience of the author, controlling the treatment of some 80–100,000 tools per week for nearly twenty years it was never found possible to harden plain medium carbon steels consistently at $\frac{3}{16}$ in. in thickness.

The lead process is best adapted for cutting-edge tools where resilience is not particularly sought after, since it is a curious thing that springs, leaf or coil, are rarely successful if dealt with by this means. Since the identical article would be perfectly satisfactory if hardened from a batch-type furnace it would appear, as is highly probable, that the very brief time-cycle during immersion in the lead does not allow of the complete absorption of the ferrite, by the austenite areas formed from the eutectoid at the Ac_1, during the passage from Ac_1 to Ac_3, and consequently, any unabsorbed ferrite is anti-resilient accordingly.

Quenching Mediums

The days of quenching in water disappeared about the same time as Mr. Harry Brearley's red-haired boy ! Its use is now confined either to low-carbon steels of the 0·28/0·35% carbon range or to certain special type low alloys such as Spec. D.T.D. 126A.

For use in connection with lead-pot hardening, oil or fused salt is most frequently employed, the latter being preferred since, in the presence of any lead oxide on the tools being treated, oil produces an extremely unsatisfactory condition which cannot easily be cleaned. The salts may consist of alkaline nitrates and nitrites mixed in eutectic proportion to give a melting point of around 140° C. Such melting point allows of usage to as low as 165°/170° C., but below this the mixture becomes pasty and allows of much "drag-out" which is not economical.

The volume of salt necessary for use as the quenching medium will naturally depend on the daily throughput. For a matter of 1,500/1,600 tools per shift 8/10 cu. ft. will suffice. There should, however, at all times be sufficient salt in the quenching tank to have a layer of solid at the bottom, for if the total bulk once becomes molten the temperature soars to 240°/250° C. and an effective and consistently uniform quench suitable for commercial production becomes difficult to obtain. For this reason it is usual to equip the tank with some suitable form of cooling device, usually forced air, which can be applied at will should occasion demand.

For liquifying the salt ready for use some form of immersion heater, either gas or electric, is employed, since bottom heating is out of the question if a layer of solid is to be retained. One of the great advantages of salt quenching is the ability of being able to raise the quenchant temperature should the work being handled be either high in carbon or manganese with the attendant possibility of added brittleness. The use of the salt at say 200° C. instead of 170°/180° C. would be sufficient to remove any such risk.

Most quenching salts are deliquescent by nature, and if out of use for any length of time pick up moisture to an extent which causes their use to be disastrous from the quench-crack viewpoint. Wet weather and the humid periods of early autumn are particular troubles in this respect. To overcome the difficulty the salt should be heated to a temperature of approximately 350° C. when all the moisture "boils" out quite suddenly and leaves the bath "dry."

Use can be made of the sharper quenching effect caused by moisture content, and when a batch of steel, difficult to harden through say low manganese, makes its appearance, the addition of only 250 mls. of water to

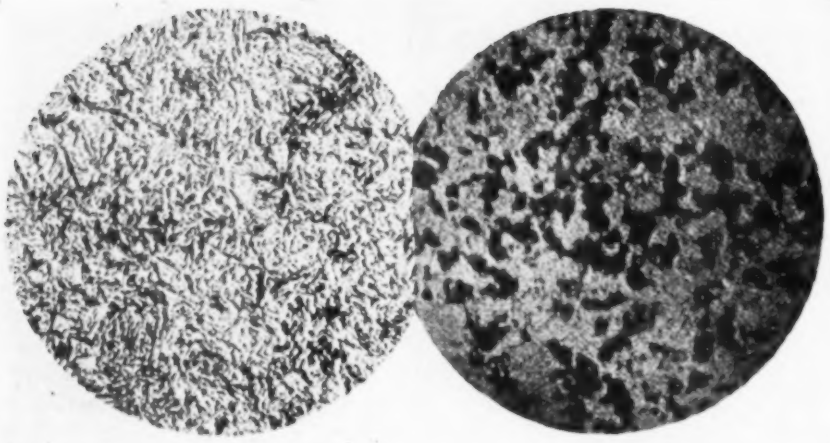


Fig. 1.—Martensitic structure obtained by quenching in a salt bath after a soaking time of 3½ mins. X 400

Fig. 2.—Troostite-martensite structure obtained by tempering the sample, shown in Fig. 1, at 240° C. for 3½ mins. only. X 400

as much as 10 cwt. of molten salt is sufficient to produce a fully martensitic structure on quenching where only a mixture of roughly 80% troostite and 20% martensite occurred previously.

Tempering from Salt-pots

Since the quenchant is mainly of salt it follows that the same medium should be employed for tempering. A separate tank adjacent is, therefore, employed containing molten salt at some higher temperature suitable for tempering probably between 280°/350° C. In this case bottom heating is employed since the temperature as a whole will not rise to such levels until the whole contents are molten. Salt of the same composition as the quenchant is used since the "drag-out" would too quickly contaminate any alternative mixture.

The tools are tempered as a "flow" production and not in batches, and again, as in the soaking time for hardening the intimate contact of the molten salt will achieve in a few minutes as much as the accepted "one hour per inch of thickness" will in a batch-type furnace.

Many readers may be sceptical of the results possible as outlined in regard to the stated soaking times for hardening and tempering, and will be interested in the two photomicrographs shown in Figs. 1 and 2. One shows the fully martensitic state obtained by quenching a plate 0·110 in. in thickness into salt at 180° C. after a soaking time of only 3½ mins., and the other shows the proportion of troostite produced at 240° C. in tempering, also in 3½ mins., this being the cycle time for a production operation on steel of 0·70% carbon.

Amalgamation of the Institutions of Mechanical and Automobile Engineers

IT was announced early in 1946 that the Institution of Mechanical Engineers and the Institution of Automobile Engineers were considering an amalgamation and that the application for the necessary powers had been made to the Privy Council. It has now been announced that authority was given by the Privy Council, and that amalgamation took effect from April 13th, 1947. On that day, the Institution of Mechanical Engineers, under the powers granted, created an Automobile Division to focus its activities with respect to automobile engineering, and also on that day an agreement between the two institutions came into force whereby the corporate members of the Institution of Automobile Engineers became corporate members of the Institution of Mechanical Engineers in appropriate classes, and simultaneously were placed on the register of its Automobile Division as the first members on the register.

This important step is the result of long and detailed deliberations by the councils of both institutions, followed by the necessary approach to the respective memberships, and throughout the stages of the consideration, certain fundamental principles have been kept in mind—namely :—

(*a*) Automobile engineering is essentially mechanical engineering—mechanical engineers should have their interests promoted by one powerful institution.

(*b*) The technical education and practical training of engineering aspirants would be better fostered by an amalgamation of engineering institutions which would provide a recognised national qualification in place of a number of specialised sectional qualifications. It would follow that the student would enter the profession through one broad entrance and subsequently specialise.

Under the provisions of the amalgamation, the Automobile Division of the Institution of Mechanical Engineers will enjoy a considerable degree of autonomous life—its affairs will be managed, under the general direction of the I.Mech.E. Council, by the Council of the Automobile Division elected by the corporate members of the Division. The Automobile Divisional Council will be responsible for the technical meetings of the Division, and its papers will be published in a separate section of the proceedings of the I.Mech.E. Local centres of the Automobile Division will be organised in the branches of the Institution.

THE Minister of Supply announces that he will cease to trade in silicon metal after June 30th, 1947. Orders for metal to be delivered from July 1st, 1947, onwards should be placed on the usual trade suppliers.

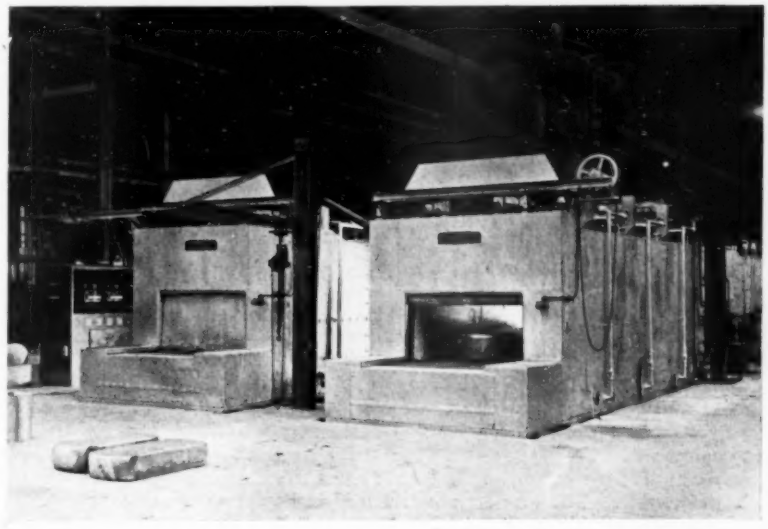
With the agreement of the President of the Board of Trade, individual licences will be issued for imports of silicon metal and applications for licences should be submitted in the usual manner to the Import Licensing Department, Board of Trade, 189, Regent Street, London, W. 1.

Heat-treatment Furnaces

Some Recent Installations

Selection of suitable heat-treatment equipment, because of the many variables affecting the quality and overall cost of heat treated products, is usually a compromise between the ideal and the practical. In this article it is possible only to indicate the trend of development by showing and describing briefly a few of the many recent installations. The need for care in the pre-heating operation is emphasised because of its affect on the quality of the component when finally heat-treated.

THE influence of heat on the quality and cost of nearly all manufactured products is very considerable, and it is not surprising that efforts are continuously being made to obtain a better understanding of the underlying principles involved and to the possibility of controlling the many variable factors affecting the conduct of heat-treatment operations. It must be remembered that heat-treatment is not solely concerned with the final heating and cooling operations to produce the properties desired in the finished product, but that the process begins with the initial operation preparatory to fabricating. This initial heating has a marked influence on subsequent heat-treatment and machining operations and on the results obtained with the finished product in service.



Courtesy of Templewood Engineering Co. Ltd.

Fig. 1.—Charging ends of two high capacity internal conveyor furnaces serving a large press.

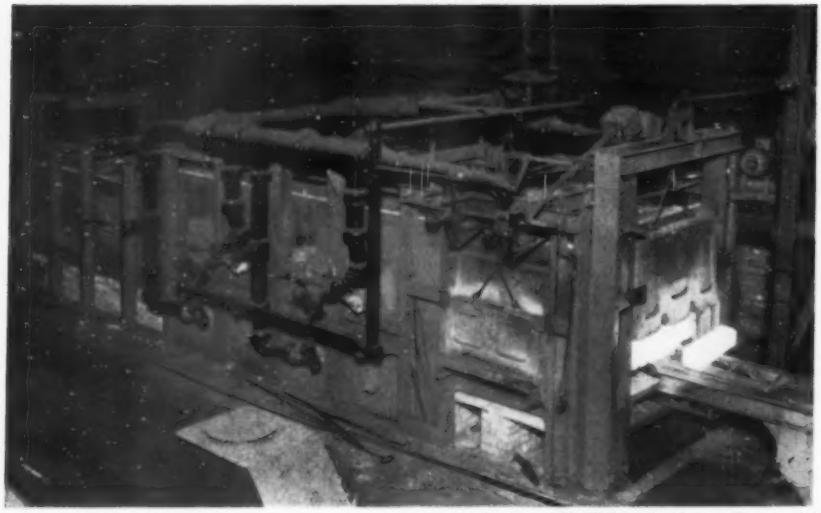
Pre-heating is very important with all metals and alloys, but it is especially important in the treatment of light alloys. Furnaces are used for heating billets prior to rolling, extending, stamping, forging, and pressing operations and for light alloys operate at about 500° C. The actual temperature employed depends on the particular alloy and may be critical, hence accurate temperature distribution and control of this type of furnace is very essential, and the correct design is greatly concerned with the elimination of all variations of temperature in time and space within the working chamber in spite of the wide range of materials and working conditions inherent in wrought metal production.

In deciding on the suitability of any furnace for a particular application it must be borne in mind that the modern furnace is a precision machine, and that its output, allowing reasonable margin for overload, is decided at the time of design, whilst the production rate of a hammer, stamp or press can be stepped up several hundred per cent. by time and motion study and by modification to die design, etc. In the heat-treatment shop, the furnaces are handled by trained furnace operators, and furnace time cycles and outputs are recognised and adhered to. In a stamp shop, the operations are primarily concerned with the hammers, the furnaces being regarded as secondary equipment, and seldom receiving the same care and attention. When hammer output exceeds furnace capacity, the furnace is blamed even though the figures may show the furnace to be performing miracles when compared with its nominal rating. Great care must, therefore, be taken both in estimating requirements and in designing to meet them.

For such large stampings as radial engine crankcases and jet engine compressor rotors, semi-continuous furnaces are most suitable. These have mechanised hearths and cold billets are charged at one end at the same rate as hot billets are withdrawn at the other, except that whereas the latter are taken out one at a time at say two minute intervals, it is usual to charge cold billets a whole row at a time, say once every half-hour, the operation of the hearth is, therefore, intermittent.

To maintain the maximum billet heating rate with uniformity of temperature, high-speed recirculation of the air inside the furnace is essential, therefore, high temperature recirculating fans force the air around the billets throughout the entire chamber and return it to the fan suction through return ducts. The heat required is added to the air during this return passage, so there is not direct radiation, all the heat being conveyed to the billets by convection ; with the temperature control arranged in the air stream before it passes over the billets, overheating of the material cannot normally occur.

It has been found advantageous in minimising certain forms of blistering of the alloys to exclude from the working chamber all products of combustion from gaseous

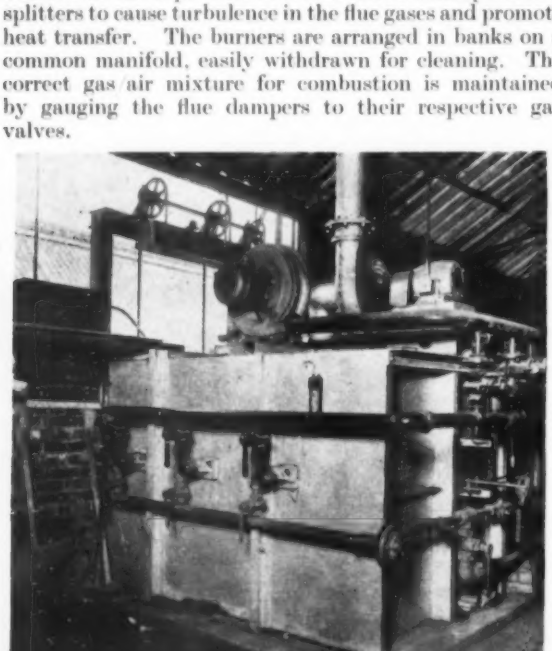


Courtesy of The Wellman Smith Owen Eng. Co. Ltd.

Fig. 2.—Continuous reheating furnace for nickel-chromium ingots.

With regard to controls, it is most important to realise that a furnace is only as good as its controlling instrument, temperature uniformity being impossible without reliable instruments. With the high rate of heating, frequent opening of doors and close control required, multizone working is essential for semi-continuous furnaces. Pyrometric instruments both controlling and recording and usually of the iron - constantan - thermo - couple potentiometric type are fitted on a spring-mounted panel in the furnace control cubicle. The latter also contains push-button control gear, fuses, isolators, etc., for the fan and hearth drives, and for safety devices which include interlocks for fuel pressure failure, excessive temperature and fan failure, all operating on the main magnetic fuel valve. An automatic gas-fuel pressure governor is also fitted.

fuels. Templewood gas-heated pre-heating furnaces are indirectly fired, the heating air is circulated by vertically mounted axial flow fans and picks up its heat from banks of specially treated, seamless steel heat-interchange tubes, carrying the products of combustion internally and arranged vertically in return air ducts within the main furnace casing. Each tube has an individual "neat" gas burner below and discharges into a common flue box at the top. The tubes are fitted with spiral core splitters to cause turbulence in the flue gases and promote heat transfer. The burners are arranged in banks on a common manifold, easily withdrawn for cleaning. The correct gas/air mixture for combustion is maintained by gauging the flue dampers to their respective gas valves.



Courtesy of British Gas Council.

Rear view of gas fired permeable-lined heat-treatment furnace.

Concerning fuels, cold, clean producer gas, towns' gas, and the like can all be used most satisfactorily in indirect fired furnaces. Oil is employed with direct firing and is efficient but less easily controlled. Electric furnaces are often favoured for the final heat-treatment operation, but for many pre-heating furnaces, where the doors are always being opened, heat losses are considerable, and even the use of the heaviest insulation cannot appreciably increase the overall efficiency.

With regard to mechanised hearths, slat conveyors offer the considerable flexibility for carrying varying shapes and sizes of billets. In some designs the conveyor leaves the working chamber and returns beneath the furnace; in the most modern the return is internal with spring tensioning of the conveyor chains at the charge end, this is claimed to be more efficient thermally, but is rather less accessible, making sound construction essential. The lightest slat conveyors can be hand-driven through gearing: in heavier models up to and including a capacity of 1 ton/sq. yd. the drive is by electric motors through a centrifugal clutch and heavy worm gear box.

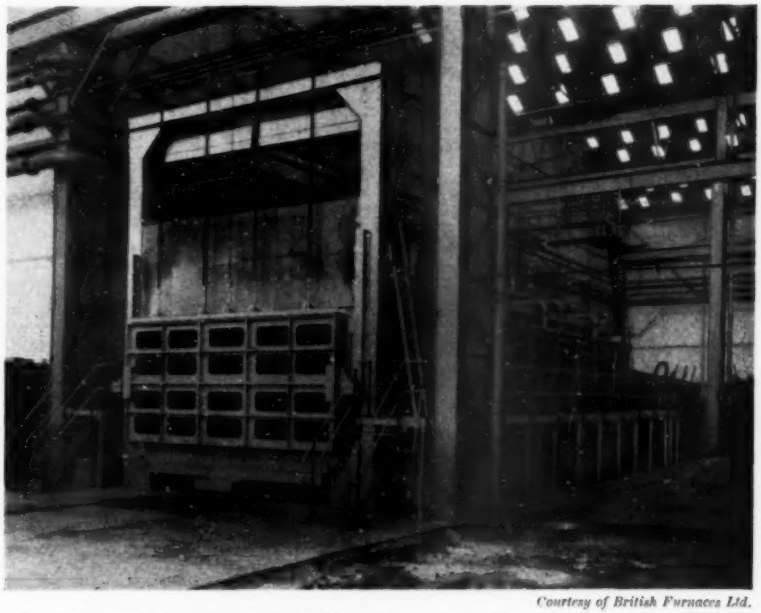
Since the billets are removed one at a time and since only very small temperature variations are permissible, it is clear that the door gear must be capable of operating quickly with the minimum effort on the part of the operator.

The main aim of the modern pre-heating furnace, states Martin,* is to produce the maximum weight of finished product at the lowest possible overall cost. Fairly low initial cost of fuel per pound heated may be achieved in obsolete furnaces, but this is only part of the story. Over-heating causes burning and blistering, under-heating causes die damage and lost time, but the modern furnace heats all the material to the right condition. The saving in scrap from this accuracy alone will reduce the fuel bill per pound of *finished* material considerably, whilst the savings occurring from increased and steadier output make "precision heating" essential for modern production conditions.

* E. J. Martin in a communication from Templewood Engineering Co., Ltd.

The pre-heating furnaces manufactured by Templewood Engineering Co., Ltd. are designed to suit individual requirements and they are constructed in such a way as to protect vital parts from the shocks and vibrations of a heavy stamp shop. In operation they are controlled so that the maximum temperature recorded anywhere in the working chamber never exceeds the higher limit of ± 5° C. of the required working temperature. The charging ends of two high-capacity internal conveyor furnaces serving a large press, are shown in Fig. 1. They are indirectly heated with towns' gas and the discharge doors are electrically operated.

A good example of a non-ferrous alloy billet and sheet bar heating furnace is shown in Fig. 2. This is a Wellman push furnace arranged for firing by town's gas and designed for heating cuprous-nickel and chrome-nickel billets and sheet bars to a correct and uniform rolling and forging temperature. The installation comprises one continuous town's gas fired furnace suitable for heating non-ferrous billets and sheet bars. The maximum output is 5 tons per hour.



Courtesy of British Furnaces Ltd.

Fig. 3.—Plate-heating furnace for plates up to 6 in. thick and weighing up to 25 tons.

As the material to be heated consists solely of alloys such as 80% copper and 20% nickel, 80% nickel and 20% chromium, 45% nickel and 55% copper special heating rates have been applied in the design of the furnace. In the case of billets 9½ in. square and 3 ft. long (80% Cu, 20% Ni), the furnace output reaches its maximum at 5 tons per hour, the stock being discharged at 1,000° C.; 3·4 tons per hour are obtained when heating 6 in. square and 3 ft. long billets (80% Ni, 20% Cr) to 1,250° C. A somewhat higher output of 4·2 tons per hour is obtained with stock 6 in. square and 3 ft. long (45% Ni, 55% Cu) which is discharged at 1,100° C. All the billet sections are heated for either rolling or forging, the furnace being designed primarily for this purpose. Sheet bars are heated at the rate of only ½ a ton per hour in accordance with the mill requirements.

In length the furnace is 35 ft. over brickwork. The inside width is 5 ft. A 28 ft. long heating hearth is provided with two non-ferrous alloy heat-resisting skid bars set at 1 ft. 10½ in. centres. These skids are raised some 6 in. above the surface of the brick hearth to permit the free circulation of gases under the material being heated. The soaking hearth is 7 ft. in length and arranged at a level 6 in. below the heating hearth with the object of causing billets to turn over when dropping off the end of the heating hearth. Evenness of heating of the stock is thus ensured before withdrawing. The soaking hearth also is provided with two dry heat-resisting skid bars, again at 1 ft. 10½ in. centres, which keep the billets about 6 in. above the hearth brickwork level. Hence, in passing through the furnace the stock does not come into actual contact with any brickwork material. Throughout the furnace the hearth has a slight downward slope from charging to discharging end, this slope being interrupted only at the junction of the heating and soaking hearths.

The furnace is fitted with 12 Basequip K gas burners. Above the stock and towards the discharge end of the heating hearth there are two burners to each side wall arranged staggered. Under the heating hearth, again towards the furnace discharge end, are four burners, two to each side. Each of these latter burners fires into a combustion chamber 2 ft. 8 in. in width, 2 ft. 3 in. in height and in length extending the full width of the furnace. The escaping hot gases from each chamber pass through three small ports, one between the two hearth skids and one adjacent to each main side wall. Over the soaking hearth there are four burners placed in the brickwork roof and firing downwards on to the hearth. The largest capacity burners are those firing over the heating hearth, slightly smaller burners being used for under hearth heating. The smallest burners are over the soaking hearth. The capacities and dispositions of the burners are designed to give a hot hearth, slow steady heating through critical temperature ranges, rapid heating where permissible, and thorough soaking of stock before withdrawal. All the burners are fitted with special valves to give fine adjustment of gas and air in order to obtain a precise degree of atmosphere control so necessary for the successful heating of alloy materials.

The furnace gases are exhausted through ports in the hearth near the charging door. These gases are collected in a flue running some 12 ft. longitudinally under the hearth on the furnace centre line. The waste products are then passed through a Newton-Needle metal recuperator and finally discharged to atmosphere by means of an induced draught chimney. The chimney is of the venturi pattern, some 40 ft. in height, the energy being supplied by an air blast from a motor-driven fan.

The fuel is town's gas specially treated to render it free from both inorganic and organic sulphur compounds, the presence of which even in very small quantities have been found to have a harmful effect on the alloys

Courtesy of The Dowson & Mason Gas Plant Co. Ltd.

Fig. 4.—A car-bottom type stress-relieving furnace fired by town's gas.

heated. Air from the recuperator is some 300° C. at the burners.

All outer refractory surfaces are heavily insulated. The furnace hearth is paved with a course of bricks chosen to resist scale attack, also to provide high thermal conductivity. These measures, together with close fitting doors and an efficient recuperator tend to give the furnace a remarkably low gas consumption. When running at the full load of 5 tons per hour and discharging stock at 1,000 °C. the overall thermal efficiency for continuous running is in the order of 54%.

The illustration is taken looking towards the furnace discharge end. Whilst the soaking hearth roof burners are not visible, gas and air piping leading to these can be seen.

A recent and very successful bogie hearth furnace, shown in Fig. 3, has been installed by British Furnaces, Ltd., for heating mild steel plates up to 6 in. thick and weighing up to 25 tons each. The area of the bogie top is 14 ft. wide × 45 ft. long, with a clear working height of 5 ft. above the top of the bogie. The working temperature is 1,100° C. and the fuel used is cold and clean producer gas under a pressure of 14 in. w.g. along with air under a similar pressure.

An individual recuperator of special design is provided for each of the burners which are arranged down either side of the furnace. These burners have a total capacity of 160,000 cu. ft. per hour, and the disposition of these combined with the flow of the gases gives absolute uniformity of temperature on the plates being heated. Automatic temperature control is arranged for by means of instruments supplied by George Kent & Co., Ltd., and furnace pressure control is also under consideration.

Stress-relieving Furnaces

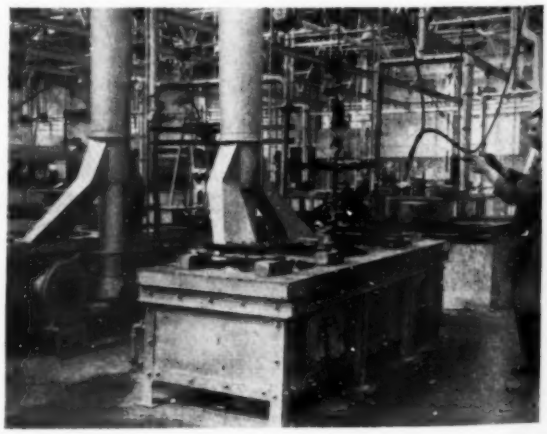
Welding applications continue to expand—particularly in directions which call for the relief of residual stresses by thermal treatment. Carbon and low alloy steels containing more than about 0·15% carbon are hardened when the zone next to the weld cools from the welding heat. The operation of heating is given to remove the brittleness and to effect a reduction of the stress by lowering the yield point at the high temperature. As the cooling takes place uniformly, no new stresses are caused.

The increasing need for this type of furnace has resulted in considerable developments in design and a noteworthy example is a stress-relieving furnace recently built by The Dowson & Mason Gas Plant Co., Ltd., and installed at the works of Messrs. Joseph Adamson & Co., Ltd. This furnace, shown in Fig. 4, is of the car-bottom type, 10 ft. 6 in. wide by 10 ft. 6 in. high from the piers on the top of the bogie to the crown of the arch, by 20 ft. long, from the inside of the door to the back wall. It is fired with town's gas and the burner equipment consists of 16 wide range, turn down gas burners, installed in two groups, half firing at hearth level between the piers on the bogie and the other half firing in the space under the arch, above the vessel being heated. This method of cross firing obviates direct flame impingement on the work and also creates a circulation of hot gases which is very desirable. A special feature is the incorporation in the side walls of recirculating ports connected with the bottom burners, which fire through venturi refractory ports.

The furnace will operate on stress relieving at temperatures between 620° and 650° C. on all types of vessels, taking less than 2,000 cu. ft. of town's gas per ton of material heated. The temperature control equipment is of interest in that the e.m.f. of the thermocouple is converted to a pressure and this is measured by a pressure controller calibrated in temperatures, which, through an air leak system, controls lever motors which are, in turn, linked with the various valves.

Carburising

In practically all sections of engineering there will always be a number of parts which are called upon to



Courtesy of The Standard Motor Car Co. Ltd.

Fig. 5—Part of a battery of cyanide pot furnaces by Kasenit Ltd.

resist wear, yet at the same time must have ample toughness and resistance to fatigue. It is a well-known fact that extreme hardness is always accompanied by brittleness, therefore, if the component is to be capable of withstanding wear, and at the same time be free from brittleness, case-hardening must be resorted to. The carburising medium may be either solid, liquid or gaseous. The use of solid carburising compounds continues to be applied to a greater extent than either liquid or gaseous mediums, however, appreciable progress has been made in the use of such mediums, and reference will be made to some recent installations.

Liquid Carburisers.—Following the introduction of the use of liquid carburisers it became quite usual for a battery of cyanide pot furnaces to be installed in the production lines at convenient points. This can be done without monopolising an undue amount of space since room is not required either for packing or unpacking of boxes or tubes, or as storage space for space-boxes, tubes or compound.

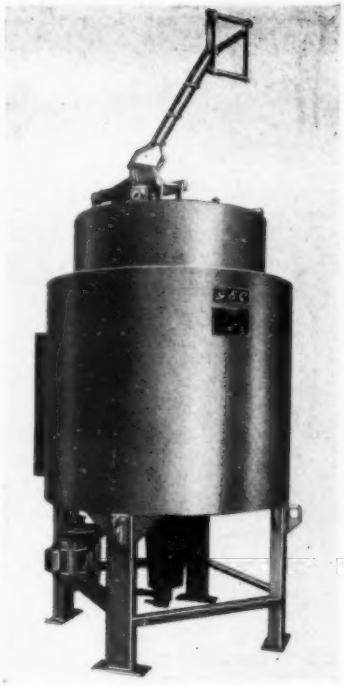
A typical installation of a battery of such furnaces, sited in the production lines, may be seen at the works of a well-known firm of motor-car manufacturers, a corner of which is shown in Fig. 5. It consists of 22 furnaces, each of the two carburising pots of which is capable of holding from 200–400 lb. of steel parts. Allowing for the different periods of immersion times needed to give the varying depths of case called for by their respective drawings, together with the variety of sizes and shapes of the components being treated, this installation will handle an output of the order of 2½–3 tons per hour.

The furnaces are by Kasenit, Ltd. and are simple but of robust design and consist of steel casings approximately 11 ft. 6 in. by 5 ft. by 3 ft. 6 in. high, so bricked internally as to provide three nests each carrying a steel pot 24 in. dia. by 18 in. deep. Cast on to the pot are two lifting eyes by means of which the pot may be lifted into, or removed from the furnace. The two outside pots are each heated by burners so arranged that the flames spiral around the pots. The waste gases are used to heat the centre pot before passing into the chimney. This pot serves as a pre-heater. Pyrometers, the thermocouples of which are provided with sheaths designed to resist the erosion of the liquid salts, are installed in each of the " active " pots, so that a regular check is kept upon the operating temperature. Some indication of the type of components treated is shown in Fig. 6.

By the use of " neutral " salt instead of an active one, these furnaces may be employed just as efficiently for the general range of heat-treatment processes.

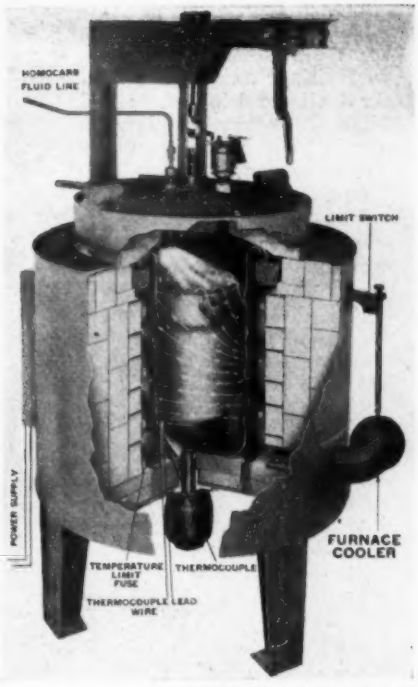
Gas Carburising.—The theory of gas carburising is not new, but advances have been made which now make it a practicable proposition in the workshop. Suitable carburising atmospheres have been developed ranging from purified town's gas to a straight mixture of air and propane. In some cases a rich hydrocarbon oil is dripped on to a plate in the furnace chamber when it " cracks " and is carried over the charge by forced circulation, induced by a fan.

Investigations have covered a wide range to arrive at the simplest and most economical atmosphere and, as a generalisation, it can be stated that if a battery of gas-carburising furnaces are to be run in one works, it is preferable to use a generator such as, for instance, a re-actor furnace in which a rich gas-air mixture is passed over heated charcoal. By this means the decarburising constituents, carbon dioxide and water, are removed. A small quantity of propane is added to the gas produced



Courtesy of General Electric Co. Ltd.

Fig. 7.—A 50 kw. top opening type carburising furnace using purified town's gas.



Courtesy of The Integra Co. Ltd.

Fig. 8.—A completely integrated carburising furnace adopting the drip feed method using a hydrocarbon fluid.

Courtesy of The Standard Motor Car Co. Ltd

Fig. 9.—A few of the components treated in the cyanide furnaces.

which makes it strongly carburising. Where a single furnace is installed for gas carburising much is to be said for the liquid drip-feed method although every application should be treated on its particular merits.

Fig. 7 shows a 50-kw. top opening type furnace by The General Electric Co., Ltd., which takes three work baskets, each 7½ in. high by 18 in. dia. The lid is of the counter-balanced lift and swing type The fan is in the hearth and drives the atmosphere over the entire charge uniformly so ensuring a consistent case depth. The gas is introduced through a pipe in the hearth of the furnace and burnt or lead away via a nozzle in the lid. It will be seen that all parts of the furnace are easily accessible.

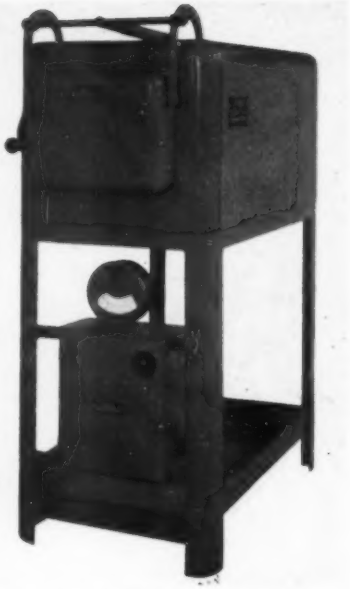


Fig. 9.–The New "Workshop" hardening furnace by Wild-Barfield Electric Furnaces Ltd.

Courtesy of Alfred Herbert Ltd.

Fig. 10.–A Thermic double-deck, high-speed steel hardening furnace.

Bell type carburising furnaces are an alternative to the above; the fan again, is in the hearth. This design is particularly useful where the charge must be cooled in a protective atmosphere, without being exposed to the air at all. In the case of the pit furnace, the baskets are lifted into a cooling chamber which is fed with an inert atmosphere during the cooling cycle.

The drip-feed method is used in the furnace illustrated in Fig. 8. This is a completely integrated equipment for carburising which not only heats the load uniformly, in a carburising atmosphere of the required consistency, but at the end of the soak it cools it uniformly until the load is ready for transfer to a cooling unit or quench tank. It is a Homocarp furnace designed by the Leeds & Northrup Company and built in this country by The Integra Co., Ltd.

A variety of gas-carburising furnaces have been developed by Birlec, Ltd.; one, designed primarily for treating small parts such as bearing rollers which can be handled in bulk, consists essentially of a cylindrical gas-tight retort, fabricated of heat-resisting alloy which is continuously rotated to ensure uniform exposure of all parts of the charge to carburising and uniform, rapid heating throughout. A modified design provides for fully automatic operation.

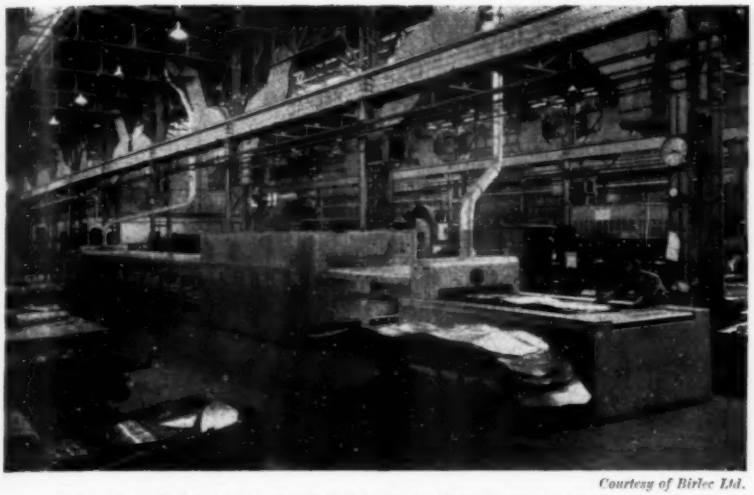
Hardening and Tempering

The modern trend towards a clean swept appearance in muffle furnaces for hardening and tempering is exemplified by the new "Workshop" furnace designed by Wild-Barfield Electric Furnaces, Ltd., as shown in Fig. 9. This equipment is entirely self-contained and occupies therefore the minimum of floor space. An energy regulator type hand temperature control panel, embodying necessary fuses and safety devices, and surmounted by a large dial-indicating pyrometer provides the means of temperature control and indication.

High-grade moulded refractory grooved bricks form the chamber and carry heating elements of heavy gauge nickel-chromium alloy designed for direct connection to 200/250 volts, single phase, A.C., supplies with a maximum rating of 8 kw. The hearth-covered with a heavy cast nickel-chromium alloy hearth tray, is 18 in. long, the door opening is 9 in. × 5½ in., and the maximum recommended temperature is 1,050° C. The door is spring-loaded to facilitate operation and is adequately heat insulated to reduce heat losses: a door switch is provided. A flue with damper is provided and provision is made for the introduction into the chamber of a suitable protective atmosphere.

An interesting two compartment furnace is shown in Fig. 10; it is a Thermic double-deck, high-speed steel hardening furnace, marketed by Alfred Herbert, Ltd., comprising a pre-heating chamber and a finish heating chamber. The furnace is fired by gas from the patent "Eddy Ray" burner, highly turbulent type, which gives an accurate mixture of gas and air resulting in very high efficiency. The nozzles of the burner are fitted with internal and external spirals which give a rotary flow to both air and gas at the point of combustion. The waste gases pass through recuperator flues before finally escaping to the atmosphere. The finishing chamber can be operated with soft-reducing atmospheres of 1,400° C. The temperature of the pre-heating chamber will be approximately 800° C. for temperatures up to 1,250° C. in the finishing chamber.

The Electric Resistance Furnace Co., Ltd. are now manufacturing the Cyclone furnace under licence from the Lindberg Engineering Company of Chicago and known in this country as the Efco-Lindberg Cyclone furnace. It is a compact unit for uniform tempering and heat-treatment at temperatures from 120°–680° C. Cyclone uniformity is due to the unique heating principle



Courtesy of Birlec Ltd.

Fig. 11.—Belt-conveyor, bright annealing furnace for copper sheets up to 4 ft. 6 in. wide.

in which gases are burned in a chamber away from the work and then mixed into a stream of rapidly circulating gases, which are driven in large volume, at high velocity through the charge. The excess volume resulting from combustion is forced out through the flue after passing through the charge. As the flue is on the suction side of the fan, no gases escape except when the burner is operating.

The gases travel from the fan into the upper distribution duct and thence into the charge. Pressure above the charge, and suction below, force air rapidly through the work basket, and the basket is located in the furnace in such a way that all gases must pass through the charge. The amount of new gases introduced to the circulating system is controlled by a thermocouple located in the upper distribution duct measuring the temperature of the gases from the fan, thus the temperature of gases entering the work basket is exactly as indicated on the control pyrometer.

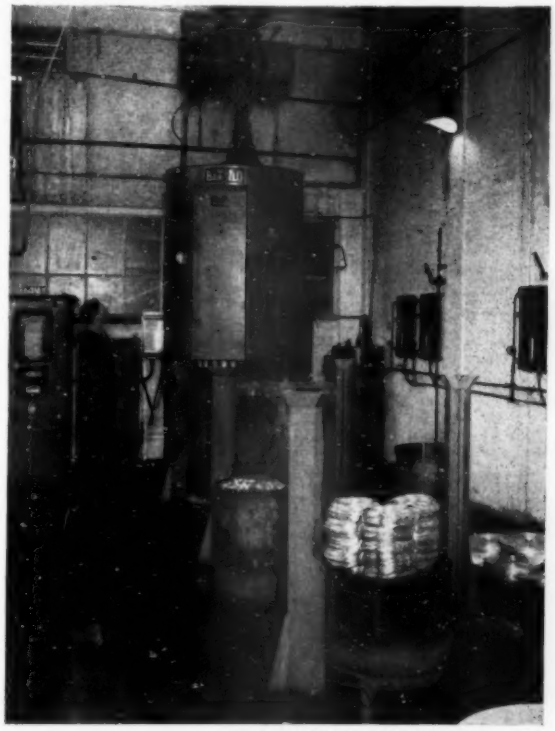
In addition to accurate production tempering, other standard uses are: Aluminium alloy solution heat-treating; aluminium alloy age heat-treating; magnesium heat-treating; nitriding (part placed in suitable retort in the work chamber, with ammonia introduced into the retort); stress relieving of welded steel parts; brass process annealing; brass stress relieving; hardening non-ferrous alloys of the precipitation hardening type such as "K" Monel, "Z" Monel and beryllium-copper.—Thus this furnace has a wide range of useful service.

Bright Annealing

One of the latest Birlec belt-conveyor type, bright annealing furnaces is shown in Fig. 11. This installation is employed for bright annealing copper sheets up to 4 ft. 6 in. wide, and has an output capacity of approximately 1 ton per hour. The conveyor belt of woven heat-resisting wire mesh passes continuously through the entrance chamber, heating chamber and cooling chamber, returning underneath the furnace. It is driven from the charging end through a patented self-compensating drive mechanism which has the important effect of maintaining a very low tensile stress in the belt at the point where it is heated. This ensures extremely long belt life, compared with that obtainable by other methods and is a very important feature in a furnace of this type.

The furnace atmosphere is prepared from town's gas. The gas is first passed through a catalytic unit which removes practically all organic sulphur compounds and is then delivered, mixed in accurate proportions with air by an automatic mixing pump, to a combustion chamber where reaction takes place. The gas is then cooled and passed to a final purifying stage where any residual H_2S is removed before the gas enters the furnace.

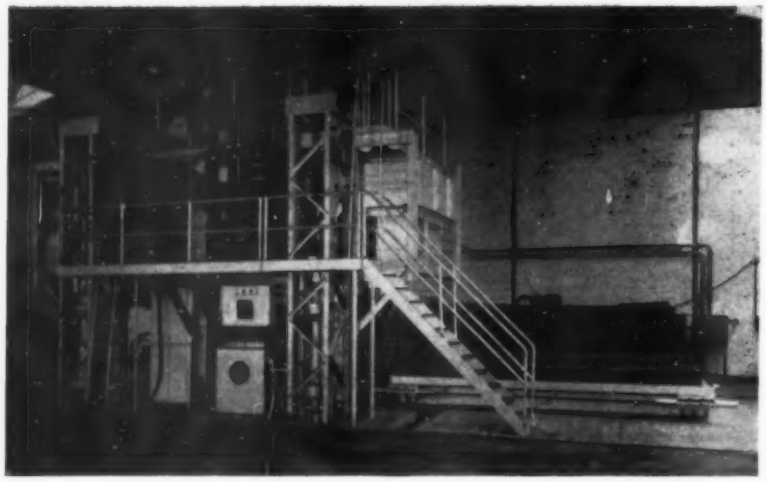
The copper sheets and circles which are annealed in this equipment are delivered in a fully-softened condition and perfectly bright. The sheets are loaded two or three high on the conveyor and travel through the process completely in about 1 hour.



Courtesy of Wild-Barfield Electric Furnaces Ltd.

Fig. 12.—A bell-type bright annealing furnace with three bases.

A recent installation by Wild-Barfield Electric Furnaces, Ltd. is shown in Fig. 12. The plant comprises a bell-type furnace with three working bases, each with its own gas-tight retort, requisite switchgear and control panel and an I.C.I. ammonia cracker. This furnace was supplied for the bright annealing of copper, brass and rare metals in a protective atmosphere. Such an installation permits one charge to be heating whilst



Courtesy of Birlec Ltd.

Fig. 13.—A gas malleabilising furnace for the treatment of white-heart malleable iron castings.

others are cooling and thus achieves continuous production although the work is actually heat-treated in batches. In order to ensure temperature uniformity of the charge and to increase the speed of heating, each base is provided with a centrifugal fan which causes a vigorous circulation of protective atmosphere through the work being heated.

Manipulation of the bell is facilitated by an electric hoist and travelling gear operated by finger-tip remote control switching.

Gas Malleabilising

The gaseous malleabilising of white-heart castings has received considerable attention during the last few years, although the theory cannot be claimed as new. The treatment, carried out at temperatures of the order of 1,050°C., is essentially one of decarburisation and/or graphitisation. The furnace must be of gas-tight construction and provided with a fan to ensure circulation of the atmosphere. Successful malleabilising has been carried out in wet hydrogen, partially burnt town's gas, etc., but work with two obvious atmospheres rendered the foregoing hopelessly uneconomical. The atmospheres referred to are air and steam.

Dealing with air, when it comes into contact with the hot iron, the oxygen re-acts with the carbon with the consequent formation of carbon monoxide. By the introduction of controlled quantities of air and the resultant formation of CO_2 it is possible to obtain an atmosphere in which the CO/CO_2 ratio is decarburising but not oxidising to iron at 1,050° C.

If steam, is used, it is introduced to the malleabilising chamber at the correct rate and a mixture consisting of carbon monoxide, carbon dioxide, hydrogen and water vapour is produced. Again, gaseous equilibria must be considered, but once more the CO/CO_2 and H_2/H_2O ratios can be quite easily kept on the non-oxidising sides of the appropriate curves, but in this case the total volume of decarburants is much higher than with the air process.

There are applications for both techniques, but in either case the speed of malleabilisation is infinitely greater than when using the hematite ore, especially on thin gauge material.

Either bell or horizontal batch-type furnaces are used for gas malleabilising and many installations have been designed and built by The General Electric Co., Ltd. The main essential, in addition to having a robust furnace to stand up to the temperature conditions, is that the correct atmosphere circulates over the charge and ensures uniformity of treatment.

A typical gas malleabilising furnace is shown in Fig. 13. It is of the elevator type—i.e., the heating chamber has its opening in the bottom and is mounted on a structure above floor level. Two alternative hearths are provided, each in the form of bogies so that either of the two can be run underneath the furnace and elevated into position, thus closing the bottom opening. The installation illustrated is by Birlec, Ltd. and is used for annealing malleable conduit fittings and similar light parts. These are placed on mild steel plates, separated by transverse joists to allow free gas circulation throughout the charge. In a furnace 14 ft. × 5 ft. × 2 ft. high, as illustrated, loads up to 4 or 5 tons can be accommodated in this way.

The bogies are elevated into the furnace by hydraulic rams, and the chamber is then made effectively gas-tight by suitable seals. By means of an automatic control, a decarburising atmosphere is maintained consistently in the furnace chamber throughout the treatment, thus bringing the material to a suitable malleable form while preserving a scale-free finish. Accelerated cooling of the charge is provided by a special atmosphere circulation system which is brought into operation at the appropriate time by the operation of dampers. The total cycle time is between 24 and 40 hours, according to the thickness of castings and other factors. The operating temperature is about 1,050° C.

MR. C. F. J. FRANCIS-CARTER, O.B.E., has resigned his post as Director and General Manager of the Metal and Produce Recovery Depots and has been appointed General Manager of the newly-formed Aluminium Wire and Cable Co., Ltd., Port Tennant Works, Swansea, and at 10, Buckingham Place, London, S.W. 1.

MR. B. WASDEN has been appointed a director of the Hallamshire Steel and File Co., Ltd., Sheffield. He has worked for the Company the whole of his business career and at present is a member of many national and local committees dealing with rolled and re-rolled steel.

MR. E. H. MCCONNELL, the Manager in Northern Ireland of the interests of George Cohen Sons and Co., Ltd., has been awarded the M.B.E. He played an important part in organising the export from Northern Ireland of scrap for use in the National war effort, and also organised the Red Cross Salvage Scheme in conjunction with the Ministry of Commerce for Northern Ireland—whereby approximately 10,000 tons of scrap metals were obtained for the war effort, and incidentally, the Red Cross benefited to the extent of several thousand pounds.

Developments in the Aluminium Industry

By Dr. E. G. West, F.I.M.

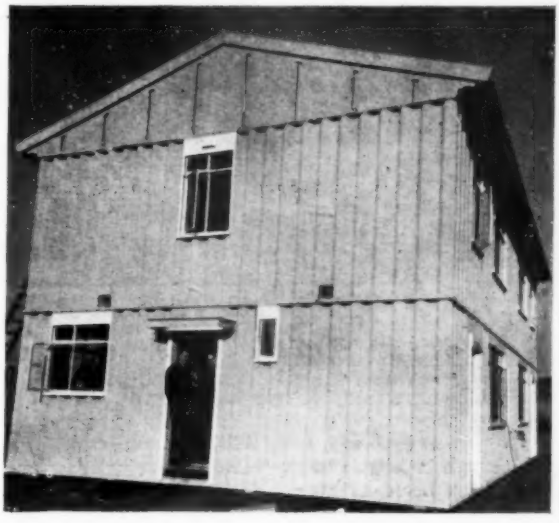
The aluminium industry, built up on a basis of scientific research and precision control, realises that only by persistent work can aluminium and its alloys be successfully applied in the great engineering industries. The better availability of aluminium has certainly permitted its use to expand, but its application in many fields is based on its fitness for particular working conditions which will assist permanent developments. Some indication of present trend is given in this review of alloy, process and application developments.

THE aluminium industry has reached a most interesting phase—one might almost say a climax—in its sixty years of commercial development and, at such a time it is more valuable to make a general survey of the situation than to concentrate on any particular aspect in detail. When it is recalled that less than ninety years ago the price of aluminium was £60 per lb., the gradual reduction to the present price of just over 8d. per lb. is a truly remarkable industrial phenomenon.

World production expanded during the war years to a maximum of some 2½ million tons annually, and present production of possibly 1 million tons a year is increasing. This total is even more impressive when considered and compared on a volume basis with the other common metals, and there is little doubt that the alteration in the relative position of aluminium and the older metallic materials is likely to be a permanent feature in world economy.

Technical advances in the industry during the war period, although important, were not spectacular, but they were sufficient to force aluminium alloys to the attention of forward-looking engineers and designers in all the pre-war using, and potentially using, industries. The development of aluminium production and fabrication has been in step to a considerable extent with the progress of the aircraft industry, and the lessons learned in co-operation with that industry may now be applied in many other fields. The wealth of knowledge and experience accumulated in the past by the users, as well as the producers, should, therefore, be applied now, while world shortage of other materials is acute. Unfortunately, there is also a comparative scarcity of aluminium and aluminium alloy in semi-finished forms despite the abundance of the raw materials, so that all present demands cannot be immediately satisfied.

The aluminium industry is certainly not complacent about the future and fully realises the tremendous amount of work that has to be completed in a comparatively short time in order to secure the full benefits possible to the industry in the near future. The fact that the industry has been built up on a basis of scientific research and precision control must be brought home to the users, especially the new and the potential users, in the great engineering industries—shipbuilding, railways, road transport and structural engineering.

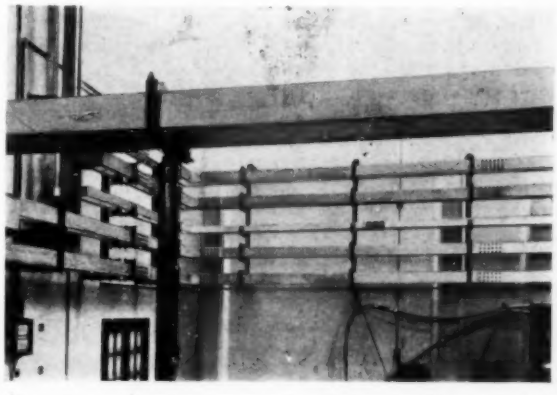


Courtesy of Rotinoff Construction, Ltd.

Fig. 1.—Prototype of aluminium prefabricated two-storey permanent house, designed by Rotinoff Construction, Ltd.

The present review is confined to three major aspects:

1. Alloy development.
2. Process development.
3. Application development.



Courtesy of British Aluminium Co., Ltd.

Fig. 2.—Aluminium bus-bars at Mount Pleasant Post Office sub-station—the relative prices of copper and aluminium have now stimulated use of the latter for electrical conductors, and this installation is typical of a number which have now seen many years' service.

Courtesy of British Aluminium Co., Ltd., & S. Schneider & Sons, Ltd.

Fig. 3.—Fixing aluminium alloy roofing (D.T.D. 213A) at S. Schneider & Sons, Ltd., London, E. 1. The estimated area of aluminium roofing is 14,000 sq. ft. and tiles or panels are 3′ × 2′ with an interlocking overlap.

Alloy Development

Although it is true to say that no revolutionary new alloys have been discovered during the past few years, considerable development in the alloy field has taken place. Perhaps the most spectacular feature is the increased use, following the increased knowledge, of the high-strength alloys containing essentially additions of zinc and magnesium. These materials, which have been covered by Specification D.T.D. 363 for some considerable time, are now in production by most of the alloy makers. Much work is still in hand on such factors as the best proportions of the alloying elements, the working properties of these alloys, their welding characteristics, and their resistance to corrosion, but the increase in the minimum Specification figures is noteworthy :—

	0·1% Proof Stress	Ultimate Stress	Elongation
D.T.D. 363 (1937)	27 tons/sq. in	33 tons/sq. in.	8%
D.T.D. 363 A(1944)	30–33 ,, (according to size)	35–38 ,,	8%

The commercial advance of alloys of this type must not, however, obscure the progress in the original high-strength alloy types and particularly in materials of the D.T.D. 364 type. This was mentioned by Aitchison[1] in his paper to the Royal Aeronautical Society in which he pointed out that the proof stress of the age-hardened duralumin-type alloy had been raised from 15 tons/sq. in. in the first world war to 26–30 tons/sq. in. in to-day's precipitation-treated alloys. The cast aluminium alloys have also shown a useful increase over the same period, Aitchison quoting the following values for the aluminium-silicon alloys :—

1919 L.33-type..	Proof stress 4 tons/sq. in.
	Maximum stress 10 tons/sq. in.
1945 heat-treated	Proof stress 16 tons/sq. in.
types	Maximum stress 18 tons/sq. in.

In this paper it was suggested that the limit of strengthening by alloying may have been reached now that aluminium alloys possessing a maximum stress some nine or ten times that of the unalloyed metal are available. This thought-provoking paper also suggested that ductility *per se* should not always be regarded with perhaps the awe or deference which the engineer is accustomed to pay. The response to this suggestion at the meeting was perhaps not quite as definite as the author might have wished, but the views expressed may well indicate a new line to be profitably followed.

Coming nearer to the immediate commercial level it may be reported that considerable progress has been made in formulating a series of British Standards for all the commonly accepted engineering aluminium alloys. It must be remembered that the products of the aluminium industry are the raw materials of many other industries, and there is no doubt that it has been a source of difficulty to many would-be users that the aluminium alloy field appeared to be so vast, whether judged by the number of proprietary alloys or by the various specifications. A useful step in reducing the number of specifications was taken when the STA. 7 Schedule of Aluminium Alloys was issued jointly by the Ministry of Supply and the British Standards Institution ; and the new series of alloy Specifications[2] for general engineering purposes, circulated to industry for comment by B.S.I., include all the well-established alloy groups from the pure metal up to the high-strength wrought and cast materials. The general issue and acceptance of the new alloy series should result in a tremendous impetus towards the full appreciation by engineers of the aluminium family, and its possibilities.

Process Development

This may be divided into two sections – namely, improvements in the processes involved in the production of aluminium and aluminium alloy semi-finished forms ; and developments in the methods of manufacturing finished goods of the aluminium-base materials.

The producing and fabricating sections of the industry have consolidated much knowledge and experience during the past few years in connection with the process of casting, rolling, extrusion, forging, etc., with the result that the degree of reliability and uniformity of product available to the consumer is higher than ever before. Alterations, and especially additions, to plant within the industry should produce better service to customers and will expand the range of available dimensions of semi-finished products.

The recent announcement[3] of a new continuous mill in South Wales indicates the importance attached by the industry to this aspect of development. The experimental production of large plates such as are required by the shipbuilding industry, clearly shows the direction in which aluminium alloy production is tending.[4]

Continuous casting of billets is a factor of major importance in maintaining and increasing the already high standard of product ; whilst improvements in foundry technique enable castings to compete in many fields, both with wrought light alloys and with the older and more established metals.

Turning to improvements in the fabrication of finished goods, welding is perhaps the field in which most development has taken place. Researches into the improvement of weldability of the medium- and high-strength alloys has not yet yielded complete answers, but at the Symposium on the welding of light alloys sponsored by the British Welding Research Association during the Autumn of 1946, interesting results were reported. The ideal of a weld metal with properties approximating to those of the wrought parent metal was clearly stated and

1 L. Aitchison. Paper presented to the Royal Aeronautical Society, April, 1946.

2 Draft British Standard Circulated to Industry for Comment, 1946.
3 E.g., *Met Ituspia*, Dec., 1946, **35** (206), pp. 59–61.
4 E.g., *Financial Times*, Oct. 14th, 1946.

progress along two lines – improving the weldability of the 7% magnesium alloy and improving the mechanical properties of wrought material of the D.T.D. 424—composition—was disclosed by B.N.F.M.R.A. investigators.[5] Commercial development along either of these lines may not be practicable at present, but the line of approach is worthy.

Another investigation in the same direction was disclosed in the three papers[6] presented at the Institute of Metals General Meeting on the effect of alloy composition on the tendency to crack during welding. Reference to the original papers in all these fields is especially worthwhile and much of the information is clearly applicable to operations other than welding.

On the side of welding processes, particular attention must be drawn to the excellent work by Tylecote[7] on pressure welding, as he has now fully demonstrated that aluminium and all the wrought aluminium alloys may be successfully joined at temperatures several hundred degrees below their melting points. Pure aluminium and the aluminium-magnesium-silicide alloys are especially amenable to pressure welding, and commercial development on the principles laid down so clearly in these researches should be immediately possible. That it may be slow in the initial stages is understandable for the procedure must be worked out experimentally for particular assemblies, and in most cases the necessary jigs and tools would require to be made before even experimental production could begin. Initially, it is likely that the process will be applied to a variety of articles such as collapsible tubes and other similar items used in the packaging industry. Looking further into the future, it seems logical that the principles uncovered by Tylecote should be used in the processes of spot welding, seam welding and butt welding to produce joints free from the "as cast" slug and the over-heated region of lower mechanical properties than those of the basis metal.

5 E. A. G. Liddiard, B.N.F.M.R.A. Report No. R.R.A. 722. J. Pen Ileton and E. A. G. Liddiard, B.N.F.M.R.A. Report No. R.R.A. 655.
6 A. R. E. Singer and P. H. Jennings, *J. Inst. Met.*, 1946, **73** (4), 197–212, and 1947, **73** (5), 273–284.
A. R. E. Singer and S. A. Cotterell, *J. Inst. Met.*, 1946, **73** (1), 33–54.
7 R. F. Tylecote, B.N.F.M.R.A. Report No. R.R.A. 673 and R.R.A. 703 and Development Report No. 46.

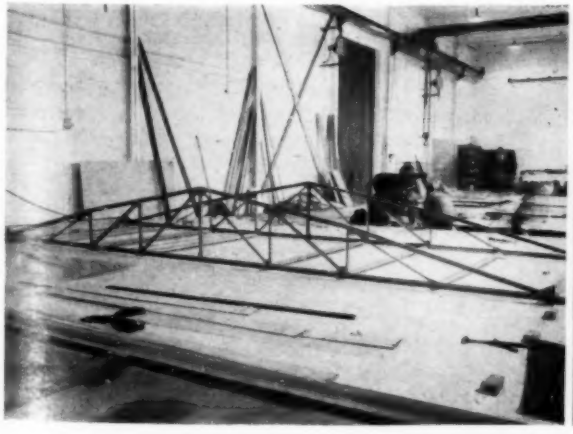
There has also been some progress in flash butt welding of aluminium alloys, but here the limitation is mainly one of suitable machines rather than lack of knowledge of the required technique. Aluminium brazing, especially by the furnace process, is also of great interest in many possible fields, but adoption of this means of joining is again slow at present.

Application Developments

Interest in applications has grown tremendously since the end of the war partly, no doubt, because of the shortage of other materials, but this interest has neither been confined to the large users nor to those possible users seeking a temporary substitute for steel, timber or other scarce materials. It is of particular value to note that many inquiries are firmly based on a long term policy.

Building.—There is no doubt that the aluminium temporary bungalow which is taking some 40% of the industry's sheet, strip and extrusion capacity has forced aluminium to the attention, not only of the architectural profession and the building industry, but also of structural engineers. The spectacular manner in which finished bungalows are handled and transported in four sections for ultra-rapid erection has shown the advantages of low weight, combined with high structural strength and stability. In the special case of the prefabricated bungalow for use in the tropics, aluminium has the further advantages that it is proof against attack by insects and will not rot, warp or swell. It is also of high fire resistance.

The fact that aluminium sheet has been used for the wall cladding and roofing of this bungalow has stimulated its application as an outer covering for several other forms of wholly or partly pre-fabricated house construction and the first all-aluminium pre-fabricated permanent two-storey house, erected in London, has received a considerable measure of approval. This house, designed and sponsored by a well-known engineering concern, is shown in Fig. 1 and embodies many novel ideas, using aluminium alloy extruded sections and outer cladding, as well as many internal items of aluminium, including staircases.

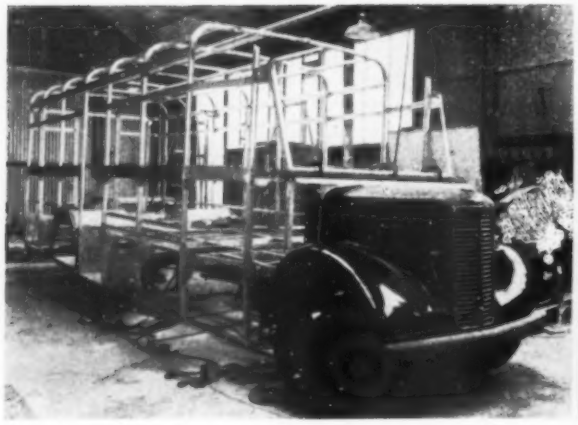


Courtesy of Aircraft Industries Research Organisation on Housing.

Fig 4.—**Aluminium alloy roof trusses joined by ceiling bearers for the aluminium house.**

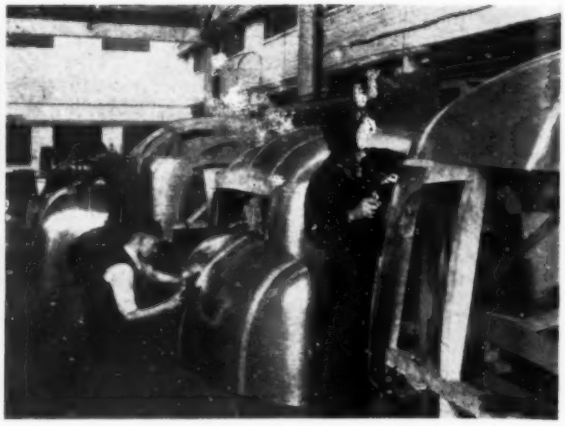
Courtesy of Air Containers (Leatherhead), Ltd.

Fig. 5.—A typical example of the use of aluminium for furniture—the utility-divan bed.



Courtesy of Thos. Harrington, Ltd., Hove.

Fig. 6. General structural assembly of an all-metal single-deck omnibus body.



Courtesy of C. H. Roe, Ltd., Leeds.

Fig. 7. Pressing and fixing aluminium panels of bus bodies.

For the more conventional type of house, aluminium has won general acceptance for rainwater goods which are now being produced both as castings and as fabricated wrought products. Work is in progress on the formulation of British Standards for aluminium rainwater goods, and building operatives have welcomed the trend towards lighter and more readily handled components in this field. Window frames are progressing from the luxury type article as used before the war for hotels and commercial premises to mass-produced casements for houses of all types. Here, several methods of fabrication have been developed, including the all-cast window, the flash-welded frame, windows with mechanical and plastic bonded joints.

Not only are aluminium alloys being used for the fittings—bolts, stays, handles, hinges, etc.—of aluminium windows, but they are very suitable for use on steel window frames, and it is to be expected that both die-cast and hot-stamped items of this kind will be used more and more extensively. Many such items are proposed for inclusion in a British Standard for building hardware.[8] Further developments in the use of aluminium for windows can be expected along two lines; namely, the production of special sections of greater rigidity than the normal types, and the design and manufacture of sliding sash aluminium windows. More than one company has already carried out interesting developments in the second direction.

Many of the other recommendations and possibilities mentioned in Study Committee Report No. 13 issued by the Ministry of Works some two years[9] ago are now well accepted by the industries concerned. In particular, sheets for roof cladding, sinks and draining boards, produced as die-castings and fabricated from sheet, ventilators, gas and electric cooker components, electrical fittings, kitchen cabinets and cupboards.

Structural Work.—In addition to small buildings, considerable attention is being devoted to the use of aluminium alloys structurally and there is little doubt that for many structures where the self weight is a large proportion of the total load to be carried, aluminium alloys possess particular advantages. Other factors which may also offset the initial higher cost of aluminium compared with steel are the lower maintenance charges and the reduced cost of transport and of handling on site, especially in difficult country abroad. Typical examples of structures for which aluminium may well prove economical are large span buildings, such as hangars; bridges, particularly moving buildings, and the floors of suspension bridges; roof trusses for structures such as school buildings which have to be produced to a standard pattern and in large quantities, and specialised applications such as the wind tunnel now nearing completion. A recently described example, of which this country may well be proud, is the door (reputed to be the largest in the world) for an aircraft hangar, the door opening measuring more than 1,000 ft. in length by 65 ft. high. This 200-ton structure is composed largely of aluminium alloy sections and aluminium-clad high-strength alloy sheet.[10]

Of even greater interest is the twin-leaf trunnion bascule bridge to be constructed of aluminium alloy over the River Wear at Sunderland, Co. Durham. This will be the first large bridge involving light alloys in the world. A less spectacular, but important application in this field, is to scaffold tubes.

Shipbuilding.—Work by W. Muckle has continued, and the principles laid down in his 1943 paper have been applied[11] to a series of vessels ranging from cross channel steamers to large Atlantic liner types. His latest calculations show that savings of fuel exceeding 10% appear possible by using aluminium alloys for stressed superstructures on some of these vessels, and a more recent paper[12] describes the way in which aluminium alloys may be applied to coasters. Although it remains for the principles and calculations to be applied in practice it can be stated that considerable interest is being shown in all parts of the world. The Aluminium Company of America are to sponsor two all-aluminium shallow draft ships for carrying ore, the larger of these ships being of some 10,000 tons capacity, and the results and

8 Draft B.S. No. 1541.

9 Post-war Building Study No. 13, "Non-ferrous Metals." Published by H.M.S.O. for Ministry of Works, 1944.

10 E.g., *Architects J.*, Dec. 12th, 1946, 431–4; *Mechanical World*, Dec. 20th, 1946, 686.

11 W. Muckle, *Trans. N.E. Coast Inst. of Shipbuilders and Engineers*, 1946, 62, 329–360.

12 *Light Metals*, July, 1946, 386–390.

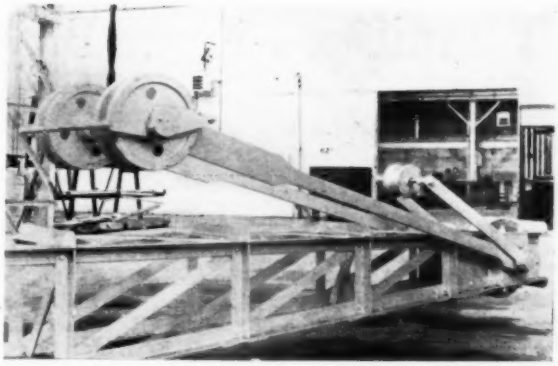
experience gained in the yards as well as in service will be eagerly awaited by shipowners and shipbuilders everywhere. There are still many problems to be solved, particularly those of shipyard handling of the aluminium alloys, including appropriate methods of preparing the surface of the plates for painting and the means to be adopted for riveting large structures.

Work is in hand on these problems and in the meantime the proved applications—lifeboats and small craft, fish holds, internal fittings and deck houses—are making steady progress in aluminium alloy.

Railways.—The railway field has always appeared attractive to the aluminium industry, but to date the aluminium alloys have not been so attractive to many railway engineers, although there are notable exceptions to this statement. Progress has perhaps been most rapid in Switzerland where many light alloy rail vehicles have operated successfully over long periods. Descriptions which have appeared in literature[13] show many novel applications, including forged aluminium alloy buffers, of which many thousands have already had a life of at least 10 years. In France, too, aluminium alloys have been widely used for railway coaches and a recent article[14] which described the effect of bomb blast on railway vehicles showed that welded aluminium alloy stock had withstood abnormal test without undue damage. Again, valuable experience has been gained in America[15] on such items as box cars and ore wagons. However, there still remain certain technical problems of design and production which must be solved before widespread use of aluminium alloys on the railways becomes general. Many of these problems are under investigation, and there are few technical reasons which would prevent the early adoption of aluminium alloy sheet and sections as structural materials for rolling stock. A start has indeed been made in this country by panelling a coal wagon with aluminium alloy and the results are so encouraging as to warrant the design of an all-light alloy vehicle.

Road Vehicles.—The advantages of aluminium alloys for all types of road vehicles are well appreciated—weight reduction resulting in better acceleration, reduced fuel consumption and increased carrying capacity: and high resistance to corrosion giving lower maintenance costs—but the various economic questions involved in changing to a new material militate against the more rapid adoption of the light alloys. The higher initial cost which is generally unavoidable when such a change is made is more readily justified in the case of goods vehicles and public service vehicles than on private motor cars, particularly the low-price mass-produced car.

The shortage of steel sheets and the difficulties of obtaining rapid delivery of cast iron components have undoubtedly stimulated interest in aluminium and its alloys for automobiles, but it must be stressed that full advantage of the change-over can be obtained only when accompanied by redesign in terms of the quite different characteristics of aluminium. Many automobile designers still do not appreciate the advantages accruing from the use of properly designed extruded sections and from die-castings. Castings afford perhaps the biggest single way of saving weight on road vehicles, and in many cases the finished component in cast aluminium alloy is no more costly than the finished cast iron component. This is due, not only to the difference in weight, but also the greater ease of machining and handling the aluminium castings.



Courtesy of Northern Aluminium Co., Ltd.

Fig. 8.—Aluminium alloy crane jib assembled with Chobert rivets.

General and Domestic Engineering.—Much progress has been made in the use of aluminium alloys, particularly castings, in several branches of the general engineering industries, in particular, for machine tools and textile plant. Several types of machine tools are now being produced with well designed aluminium alloy bases incorporating new features and such plant is shown to advantage where machine shops, etc., have to be located on upper floors of existing buildings. Furthermore, the ease of handling is a small but important factor of interest to the plant engineer.

Many parts of printing and textile machines operate with a reciprocating motion, and advantage can be taken here of the reduced inertia when the weight is halved by using aluminium. Further, aluminium is proving a very satisfactory alternative to wood for textile equipment as it is not subject to shrinkage and warping and, furthermore, it is completely non-staining.

The re-equipment of the coal industry is of particular interest for the characteristics of aluminium and its alloys make the change over to these materials attractive to the mining engineer. Experience with coal tubs, cages and skips: conveyer and ventilation components: cutters and drills has been satisfactory and the economic case may now encourage the large-scale application of aluminium to these machines and plant.

The electrical industry is also taking a great interest in aluminium, which has already been used very widely for transmission lines and bus bars. Its application to underground cables seems to be a matter of time and economics.

Finally, it is worthy of note that many well designed articles of aluminium alloy were chosen for inclusion in the Britain Can Make It Exhibition—furniture, toys, domestic machinery of all kinds, garden and sports goods. The output of aluminium furniture is now appreciable and includes beds and suites, chairs and tables, kitchen cabinets and bar stools. Castings and wrought alloys are used, with welded, bonded or mechanical joints.

In conclusion it is again stressed that the intrusion of aluminium into new fields is being increasingly based on its fitness for particular working conditions—rather than upon its better availability in a time of scarcity—clearly, the best if not the only foundation for long term permanent developments.

13 E.g., *Light Metals*, Feb., 1945, 70–78.
14 M. Guerguin, *Revue de l'Aluminium*, Aug.-Sept., 1945, 54–9.
15 *Revue de l'Aluminium*, Oct., 1946, 321–333; *Light Metals*, Jan., 1947, pp. 5–14.

Titanium—Some Properties and Applications

By H. W. Greenwood

(*Powder Metallurgy Limited, London*).

The field for high-titanium alloys is still in its infancy and is begging to be explored.

IN a F.I.A.T. Report published last year, there occurred a sentence that should be borne in mind by all metallurgists. "The field of high titanium alloys is still in its infancy and is begging to be explored." Indeed, it is not only the field of high titanium alloys that has been neglected, the whole subject of the industrial use of titanium has been dealt with in a very perfunctory style when it has received any attention at all

We hear titanium spoken of as a rare metal, yet it is more abundant in the earth's crust than any commercially used metal other than aluminium, iron and magnesium. It is some 60 times as abundant as copper and over 100 times as abundant as such metals as zinc, lead, tin, etc. Nor do we hear it spoken of as a light metal, yet its specific gravity is only 4·5 as against 7·9 for iron, 8·9 for copper, 7·3 for tin, and 11·4 for lead.

Titanium products have found wide application in industry, as witness the fact that several hundred thousand tons of titanium minerals are mined every year and used in the paint, welding and other industries, to the development of which titanium compounds have made notable contribution, but on the metallurgical side there is a tremendous field awaiting development and investigation. Titanium metal has a melting point of about 1,725° C. It is a hard metal having a Brinell hardness of about 180. In the annealed state, it has a tensile strength of 82,000 lb./sq. in., a yield strength of 62,000 lb./sq. in., and an elongation of 28%. In the cold-worked state, its tensile strength is 126,000 lb./sq. in. and yield strength of 100,000 lb./sq. in.

It forms extremely hard carbides and nitrides, also a long range of alloys having very varied and very notable properties; it can be used for cladding metals, and flows over copper surfaces to provide a titanising coat of copper-titanium eutectic, having very valuable properties. The same continuous coat of titanium can be applied to iron and nickel if they are copper-plated beforehand.

Titanium metal has many notable properties, in particular it has a high electrical resistance, some hundred times that of copper and has been used as resistance at high temperatures, higher, in fact, than are possible with nichrome. Again, titanium when alloyed with certain of the non-ferrous metals can provide age-hardening alloys of high tensile strength which is retained at temperatures much higher than those permissible for ordinary bronzes. Such alloys carrying from 1-3% titanium, balance copper or nickel, give tensiles upwards of 100,000 lb./sq. in. It is also notable that alloys containing titanium have been successful in standing up to the stresses and temperatures to which blades and buckets of gas turbines are subjected. It would appear that there is here a field that will bear much closer investigation with great advantage.

The rôle of titanium in iron and steel is not as yet fully understood, we are told that it can act as a stabiliser. In steel, it does so act, that is generally recognised, but in cast iron it has an effect which appears quite contrary —namely, that of a graphitiser. This apparently anomalous behaviour has caused it to be asked—"What does titanium stabilise?" The question is an important one, and its correct and full answer will materially advance ferrous metallurgy and particularly our control over the effect of residual elements in cast irons. Its action as a stabiliser in steel resulted in the Germans making use of it particularly for the enhanced resistance to high temperatures which titanium-containing steel showed. Another application of titanium was the preparation of copper, cobalt and nickel bronzes, containing titanium which were rolled into steel surfaces to provide a cladding which notably enhanced resistance to high temperatures.

Another German application of titanium was in the production of a light armour plate, for which they incorporated titanium into the steel surface. It was found that sandwiches of such titanium-coated plates had good armour properties. Simple steel sheets were dusted over with titanium in powder form and then heated in a hydrogen atmosphere to obtain a bond.

Experiments were also in hand dealing with the addition of about 10% titanium to cast iron, which increased hardness to an extraordinary extent.

The rôle of titanium in non-ferrous alloys has already been mentioned, particularly the production of age-hardening copper alloys. In this connection it is of interest to note that the mechanism of the process is similar to the age-hardening of copper-beryllium alloys, but the hardening is not so intense. As is the case with beryllium copper, both the hardness and the electrical conductivity of the alloys is enhanced by tempering after quenching. *Nickel and its alloys can also be hardened in the same way. A number of patents have been taken out for permanent magnets having much higher coercive force than Alinico and permanent magnet steels containing titanium.*

Titanium has another rôle in metallurgy, that of grain refiner, and is used particularly in aluminium and light metal alloys to the extent of 0·1-0·2%. Such an addition is sufficient to refine the grain so that an increase of some 15-20% in strength is attained.

Titanium for use in metallurgy is available in three forms, as the metal powder, as the hydride and as master alloys. The powdered titanium is usually used where simple cladding is called for, the hydride is the most convenient for titanising, for deoxidising and grain refining and for the protection of certain alloys, while the master alloys are best for producing the age-hardening alloys of copper, nickel, etc. Titanium in any, or all, of these forms can be obtained from Powder Metallurgy, Ltd., Commonwealth House, New Oxford Street, London, W.C. 1 who are the agents for Great Britain for Metal Hydrides Incorporated, of Beverly, Massachusetts, U.S.A. Powder Metallurgy, Ltd., will be only too pleased to answer any inquiries and to give further information as to the application of titanium and its alloys and compounds that may be required.

Some Aspects of Hot Hardness Testing

By K. G. Robinson, B.Sc.

With the increasing demand for alloys to withstand high temperatures there is need of testing techniques which provide information regarding the possible performance of new materials at elevated temperatures. An important property in this connection is hot hardness, about which some confusion has arisen due to lack of standardisation of testing techniques, and it is the testing of this property which is discussed.

THERE is an ever increasing demand for metallic alloys for use at elevated temperatures, this demand being greatly intensified during the war years with the development of the gas turbine and similar machinery operating at high temperatures, thus imposing increased requirements upon testing techniques. It is obviously desirable to have information about the performance of new materials at elevated temperatures and invariably the question arises as to the hardness of such materials at the operating temperatures, as this property very often gives an idea of the general properties to be expected of the alloy at the temperature in question. Although the determination of hot hardness would appear to be a relatively simple operation, much confusion has arisen in the past due to the lack of standardisation of testing techniques, and very often published data has not been strictly comparable owing to the different procedures adopted by the different investigators.

One of the first investigators to undertake an investigation of the hot hardness of metallic materials was J. A. Brinell,[1] who used a cold ball indentor for test temperatures up to about 330° C. The procedure adopted was very similar to that used in ordinary hardness testing, consisting of heating the specimen to the test temperature, withdrawing from the heating furnace, and then proceeding with the test in the normal manner. Although providing much valuable information at the time, this method is open to many objections. To begin with, the indentor itself is quite cold, and therefore chills the sample in the vicinity of the contact area, while the specimen under test is losing heat to its surroundings from the time it leaves the heating furnace. If an ordinary steel ball is used as the indentor at elevated temperatures, it may be softened and deformed during the course of the test. All these factors lead to errors in the determination of the hot hardness. In 1921, d'Arcambal[2] published his work on hot hardness testing of high speed steel in which he attempted to overcome some of the difficulties mentioned above by heating both the indentor and specimen; the tests were continued to temperatures as high as 600° C. Use was made of ordinary steel balls and efforts made to overcome errors due to deformation of the steel ball by using a new ball for each test, but this procedure is not satisfactory for hardness testing at the higher temperatures. It has been suggested[3] that, in using ordinary steel balls for hot hardness testing, the test temperature should not exceed the range 200–500° C., depending on the composition and resistance to tempering of the balls and the hot hardness of the material being tested, although the lower figure would appear to be the safest. Even further, it is recommended in B.S.1094, 1943,[8] which covers the hot hardness testing of light alloys in the range 100–450° C., that a new ball should be used after every test above 200° C.

The introduction of the sintered carbide ball has greatly increased the scope of static hot hardness testing, removing many of the objections associated with the ordinary hardened steel indentors, and has enabled much more accurate results to be obtained at the higher temperatures, as the sintered carbide balls retain their hardness at comparatively high temperatures. This means that it is possible to heat the indentor to the test temperature prior to making the indentation, without any danger of damaging the ball, provided certain precautions are taken. The use of tungsten carbide balls for hot hardness testing at temperatures as high as 800° C. has been reported.[4] If tungsten carbide balls are heated for a short time in air to the higher temperatures an oxide film forms, but provided the test is made as soon as the indentor reaches the operating temperature no great error is introduced, nor is any great damage done to the ball, although the number of tests which can be made with one ball is limited owing to the fact that the ball must be cleaned after each test, thus leading to a very small reduction in diameter, which is negligible however until a fair number of tests have been made. Undoubtedly, the use of a controlled atmosphere, either reducing or inert, in the furnace during preheating and testing is a very desirable refinement which leads to increased ball life and the maintenance of a better surface on the ball. Apparently American workers have made considerable use of such indentors for hot hardness testing and found them very satisfactory.[5]

Impact testing has been used very extensively by the Germans for making hardness tests at elevated temperatures and certainly overcomes many of the difficulties associated with static hardness tests at very high temperatures. In impact hardness testing, the principle involved is similar to that of such static hardness tests as the Brinell and Vickers in that in both cases an indentor pierces the test piece, causing an indentation of size related to the load applied and the hardness of the material under test, the size of the indentation being a measure of the hardness of the material under test. Using a similar principle hardness determinations have been made at elevated temperatures by the measurement of the height of rebound of a hammer which has fallen through a known height, but this has not found much favour. The usual impact test differs in that instead of using a slowly applied load as in the static test, it depends upon a falling

1 J. A. Brinell. Researches on the Comparative Hardness of Acid and Basic Open Hearth Steel at Various Temperatures by Means of Ball Testing. *Journ. Iron and Steel Inst.* 1905.

2 C. H. d'Arcambal. Hardness of High Speed Steel. *Chem. and Met. Eng.* 1921, v. 25, pp. 1168–1173.

3 O. E. Harder. Hardness Testing at Elevated Temperatures. Metals Handbook.

4 V. Ehmcke. Influence of Nickel and Manganese on the Properties of Tool Steels. *Arch. f. d. Eisenhuttenwesen.* 1930, pp. 23–25.

5 J. H. Hruska. Hot Hardness. *Iron Age.* July 15, 1937, pp. 30–34.

weight, or a blow by a hammer or pendulum. As mentioned previously, the static hardness test at elevated temperatures suffers from certain disadvantages peculiar to this type of test such as scaling, deformation and tempering of the ball and chilling of the test piece by the ball, although the latter may be overcome by preheating the indentor subject to temperature limits set by the materials used for the indentor. In addition, in the static hot hardness test, consideration must be given to creep of the test piece under load, the latter being contributory to discrepancies between the static and impact types of test. In certain applications, particularly at very high temperatures, the impact type of test overcomes these difficulties to some extent due to the brief period of contact between the indentor and the hot test piece. It is claimed[6] that the impact test gives values more closely related to actual conditions met in metal working processes. Sauerwald[7] established that, with rapid deformations frequently met in metal working processes, considerable work hardening occurs at temperatures higher than the annealing temperature required to release cold working effects, which explains one of the reasons for the different results obtained from impact and static tests, as the latter allows time for continuous recovery from work hardening effects. With impact testing, the elastic properties interfere in the case of hard materials with small indentations, when the elastically deformed region is large in comparison with the permanently deformed.[6] Another disadvantage of impact testing is the lack of standardisation amongst the various investigators so that it is difficult to compare quantitatively the various results obtained, which is a serious drawback when compared with indentation methods based on such old established methods as the Brinell hardness test, even making allowance for variations in the application of the latter types of test at elevated temperatures, as at least these types of static test have the advantage of a hardness scale recognised universally.

In order to obtain strictly comparable results from every hardness test made by any particular type of testing technique care must be taken to standardise the conditions of testing throughout. When making hardness tests at elevated temperatures, the time required in reaching the test temperature and the time at this temperature must be taken into account unless the sample is previously thoroughly annealed or tempered at a temperature not less than the testing temperature, when the sample should be in a state of equilibrium at this temperature so that time of holding at the test temperature is then of little importance and will have little effect on the hot hardness of the material. Alternatively, a standardised technique may be adopted in which the sample is heated only long enough to bring it uniformly to the test temperature and proceeding with the test regardless of whether the sample has reached the ultimate state of equilibrium as the latter in many cases takes a considerable period of time. Obviously in the latter testing technique, the time factor is of considerable importance, and to effect comparable tests the time factor has to be standardised. Harder[3] quotes some work by Wissler and Day which illustrates this point admirably; they made tests on high speed steel of the 18 : 4 : 1 type at 700° C. in which the specimens were held for 20 minutes, 1 hour, and 1½ hours at the test temperature and gave values of 275, 193 and 175 respectively, on the Brinell scale. Harder suggests that the lack of consistent results at high test temperatures among the various investigators is often due to the fact that the times of soaking at the test temperature have, in many cases varied considerably. Standard conditions for the hot hardness testing of light alloys have been proposed by the British Standards Institution[8] in B.S.1094, 1943 and covers the temperature range 100–450° C. for cast and wrought materials. This specification requires a temperature control of ±1·0% of the temperature in degrees Centigrade or ±2° C., whichever is less. The question of time is standardised by requiring that the specimen shall be brought gradually to the test temperature in 60 ±10 minutes and then maintained at the test temperature for 60 minutes before making the impression. It is also required that the indentor shall be heated and maintained at the test temperature. In considering the time of application of the load it should be remembered that much depends on the material being tested, and the test temperature, and in most cases the usual time of loading are satisfactory.

In conclusion, it would seem that for most work, the static hot hardness test gives the most satisfactory results subject to a maximum temperature of about 200° C. for ordinary steel balls, and about 800–900° C. for sintered carbide balls. This method also has the advantage of relative ease of application and hardness values may be readily expressed in Brinell or similarly widely accepted quantitative units, eliminating the necessity for converting the results of other tests into these already well-established units. For tests at temperatures beyond the range where sintered carbide balls are usable, the impact test has much to recommend it.

6 E. Fetz. Dynamic Hardness Testing of Metals and Alloys at Elevated Temperatures. *Trans. Amer. Soc. for Metals.* Dec., 1942, Vol. 30, pp. 1419–1462.

7 F. Sauerwald. Die Warmverformung der Metalle, ihre Kennzeichnung und ihre Beziehung zur Kaltverformung. *Metallwirtschaft.* Vol. 7, 1928, pp. 1355–1359.

8 B.S.S. British Standard Specification for Short-Time Testing of Light Alloys at Elevated Temperatures. B.S.1094, 1943.

The Aluminium Development Association

THE above Association has published a 16-page booklet giving its aims, activities and organisation. This Association is a technical and consultative body, voluntarily sponsored and supported by the aluminium industry of this country, with the object of encouraging the correct and increasing use of aluminium and its alloys. It co-ordinates effort and disseminates information for the mutual benefit of supplier and user, but takes no part in the trading activities of its members. This brochure reveals the many directions in which the Association's activity is given, not only directly to industry, but by co-operation with Government Departments and other technical bodies such as The British Standards Institution. In it are summarised the major activities of the Association, those already undertaken and others planned for the immediate future. Readers who are in search of knowledge of aluminium are invited to avail themselves of the facilities the Association possesses, but as a preliminary it would be useful to obtain a copy of this well-prepared brochure. Copies are available on application to The Aluminium Development Association, 33, Grosvenor Street, London, W. 1.

Modern Methods of Gas Analysis

Part IV.—The Ambler Portable Apparatus

By W. D. Vint

Many prefer the Ambler portable apparatus for gas analysis because of its greater simplicity and the fact that there is less possibility of gas leaks. Two models are in use, the works model and the precision model. Both are described, and the technique used for obtaining reliable results is discussed.

THE principle underlying the Ambler portable apparatus is different from that of the Orsat or Hempel. Aqueous reagents are still retained for the absorption of constituent gases, but instead of separate vessels for each reagent, one vessel only is used, and the particular reagent required is added to the vessel, gas absorption made, the vessel washed out, and the next reagent added. Combustion also is carried out in the same tube which is provided with a platinum spiral heated from an electric battery or step down mains transformer. From the point of view of simplicity the Ambler is a great improvement on most other types of apparatus. There is a complete lack of rubber connections with their possible sources of gas leaks, the only possible source of gas leakage being from the single ground glass tap at the top of the graduated tube. A diagramatical sketch of the apparatus is shown in Fig. 1.

Gases are measured into the 10 m¹. gas burette A and passed into the absorption vessel B which has previously been filled with reagent from the point "x". Owing to the small volume of the gas and the fact that each reagent is fresh and in small quantities, absorption is extremely rapid and errors due to physical absorption of gases in the reagent as present in other types of apparatus are totally absent. Vigorous agitation of the reagent can also be made, and as the confining liquid is mercury, no absorption of the gas in the latter is possible.

The apparatus is designed for most industrial gas analyses both simple and complex and for accuracies to within 0·1%.

Gas Absorption Reagents

1. *Carbon dioxide.*—40% potassium hydroxide solution.

2. *Oxygen.*—20% aqueous pyrogallol solution. Two ccs. of the 40% KOH is introduced into B and followed by approximately ½ cc. of pyrogallol solution. The part at "x" must be wiped dry of pyrogallol solution otherwise the latter will become contaminated with alkali and does not then keep so well. Pyrogallol is an unsatisfactorily slow absorbent below 15° C., so if it is not practical to work above this temperature, sodium hydrosulphite with sodium anthraquinone-B-sulphonate may be added (Ref. Fieser, "J. American Chemical Society," 1924, 46, 2639, and "Technical Gas Analysis," Lunge and Ambler, page 98). Further details of the absorption of oxygen by pyrogallol and other reagents may be obtained from "Technical Gas Analysis," pp. 95–103.

3. *Unsaturated hydrocarbons.*—Fuming sulphuric acid (10% oleum). Reference to Technical Gas Analysis gives further details as regards the absorption of unsaturated hydrocarbons.

4. *Carbon monoxide.*—A stock bottle of cuprous chloride solution is prepared by desolving 250 grams ammonium chloride in 500 ccs. water, adding 200 grams of cuprous chloride and finally adding water to 750 ccs. The solution should be allowed to stand 24 hours and must be shaken before use. When required, three parts of this solution are mixed with one part of concentrated ammonia, and this mixture is then added direct to the apparatus.

An absorption is not complete in one treatment; two or even three successive quantities of ½–1 cc. of ammoniacal cuprous chloride must be used. After absorption is complete the gas must be washed with ½ cc. of water to remove any ammonia gas.

Combustion

Hydrogen and carbon monoxide with an excess of oxygen are burnt in the vessel B by maintaining the platinum wire at dull red heat for one minute. Methane requires a bright yellow heat for two minutes. It is preferable to burn the combustible gases rather than explode them, and it should, therefore, be arranged that higher percentages each of 10% hydrogen or 6% in the case of methane are not present in the combustion vessel. If the gaseous mixture does contain higher percentages of methane or hydrogen, part of the sample must be expelled in a similar manner to the technique adopted on the Orsat and calculation based on the reduced sample. Should gases of high proportions of methane and hydrogen (e.g., coke oven or town gas) be regularly encountered, an explosion bulb containing a spark gap instead of a platinum spiral can be used, but under no circumstances should the proportion of hydrogen exceed 20% or methane 8·5% (intermediate figures for gases containing both) since considerable error may arise on account of oxidation of nitrogen. For the same reason there is seldom any object in adding oxygen for the combustion instead of air since there is usually enough for all the oxygen required to be added in the form of air.

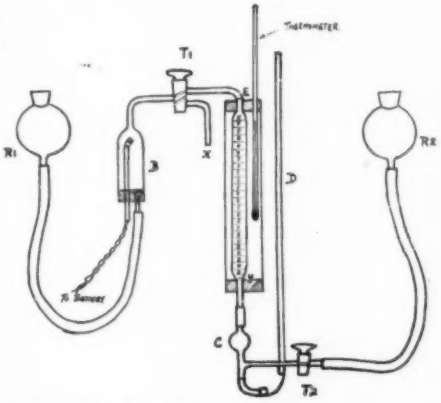


Fig. 1.—The Ambler portable gas analysis apparatus.

There are two models of the apparatus: (1) The works model which is minus the manometer; and (2) the

precision model. On the works model the gas is measured by volume at a constant pressure, whilst on the precision model which possesses a greater accuracy, gas measurements are made at constant volume and variable pressures.

Constant pressure type.—The measurements are made at atmospheric pressure by bringing the reservoir of mercury to the same horizontal plane as the level in the gas burette and taking the reading of the latter level. Accuracies greater than 0·5% of the total volume cannot be expected from this method, the limit being the approximate way in which the gas is brought to constant pressure.

Constant volume type.—The maximum accuracy of measurement is obtained by bringing the mercury to the mark "y" on the constricted tube below the gas burette and measuring the pressure by means of the manometer. For gas mixtures the analyses of which do not involve volume changes (on absorption or combustion) greater than approximately 20% of the total volume, the length of the manometer is sufficient for this method to be used; calculation of the results is as follows:—

Suppose "y" is the manometer reading when the gas burette is full of air at atmospheric pressure (this figure can be determined once for all by removing tap "T1" and bringing the mercury to the mark "y" and reading the manometer). Let "z" be the manometer reading when the burette is filled with the original gas sample with the mercury on the mark "y." Then "P," the pressure in the burette equals b + z − y where "b" is the barometric pressure. Since the burette is washed out with water before the analysis the gas is saturated with water vapour, hence, the net pressure of the gas is b + z − y − w where "w" is the vapour pressure of water at the temperature indicated by the thermometer in the water jacket.

Suppose "V" is the volume of the burette filled to the mark "y," then the volume of the gas sample reduced to 76 cms. pressure is:—

$$\frac{V(b + z - y - w)}{76}$$

If "z1" is the manometer reading after the removal of one gaseous constituent, then the volume of that constituent is:—

$$\frac{V(z - z1)}{76}$$

and the percentage of the constituent is

$$\frac{z - z1}{b + z - y - w} \times 100$$

The quantity b + z − y − w representing the original net pressure of the sample is thus determined once for each analysis, and the percentage of each constituent is directly proportional to a difference between successive manometer readings.

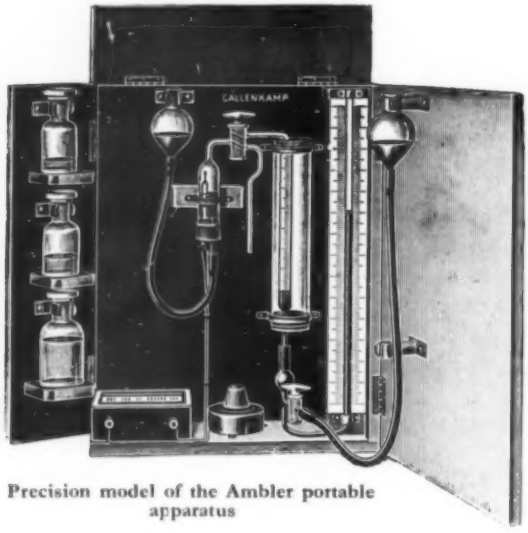
If the quantitative composition is such that the above procedure is not possible, the same general procedure may be followed, but with the range extended by using two or more of the graduation marks on the burette instead of "y." The accuracy is slightly less on account of the error involved by bringing the mercury level to a wider portion of the burette. If after one or more absorption, the mercury can no longer be brought to the original mark it is brought to some other mark in the range of the manometer tube. Suppose then, for example, the gas had a pressure "P" (calculated as above) at 10 ml. volume and after absorption of one constituent had a pressure of "P1," at 5 ml. volume, the percentage of the constituent in question would be:—

$$\frac{P - \frac{1}{2} P1}{P} \times 100$$

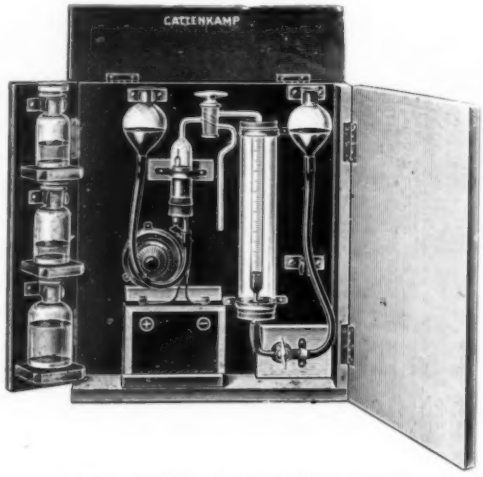
There is yet a third method of gas measurement—viz., by varying volume at approximately constant pressure. The gas is brought to approximately atmospheric pressure (within about 0·2 mm.), and the volume is measured and corrected to exactly atmospheric pressure by means of the manometer reading.

The manipulation is as follows:—

After the approximate levelling has been made, T2 is closed and the burette and manometer read. The pressure in the burette is then equal to that of the atmosphere plus the difference between "z" the manometer reading and "z2" the manometer reading when the burette is filled to the same volume at atmospheric pressure. The figure "z2" is obtained by taking volume and pressure readings at two or three different positions with T1 removed as described above. If this difference be called dP cms. the corrected volume V1 (that is the reading which would have been observed if the mercury



Precision model of the Ambler portable apparatus



Works model of the Ambler portable apparatus

had been levelled to exactly atmospheric pressure) is given as follows :—

Let "b" be the atmospheric pressure, "V" the burette reading, and "w" the vapour pressure of water, then,

$$V1 = V \times \frac{b - w + dP}{b - w}$$

$$V1 - V = V \times \frac{dP}{b - w}$$

If P is not greater than 1 cm., any deviation of "b – w" from the approximate figure of 75 cms. will not affect the correction sufficiently to introduce an error of more than 0·1% of the total gas. In practice it is found that dP does not exceed 2 mms.

The magnitude of the correction then per 1 mm. P is :—

$$V \times \frac{0{\cdot}1}{75} = V \times 0{\cdot}00133$$

i.e., 0·13% of V.

Method of Manipulation

The sample for analysis is transferred to the gas burette "A" at the point "x." Preferably, the sample should be previously contained by a vessel fitted with a three-way capillary tap. The common end is connected to "x" (glass to glass joints) and air driven out of the connections by means of mercury from "B." In the case of an air analysis, the sample may be drawn in by simply lowering R2. If flue gases are to be analysed, the sampling pipe may be connected to "A" through a three-way tap and the connections cleared of air by filling the burette with gas and expelling it through this tap, and repeating the process three or four times.

The gas is now transferred to the burette "A."

Constant volume method.—If the constant volume method is used, the reservoir R1 is lowered sufficiently to bring some gas into the bulb "C" below the burette (such that when compressed to the mark "y" it assumes a pressure of approximately 1 mm.). The gas sample is then followed up by mercury as far as the mark "E" and the tap "T1" closed. The burette is now sealed against either leak out of gas or leak in of air during the volume or pressure measurement. The method of bringing the mercury to a mark is most conveniently done by placing "R1" in the upper of the two clips provided for it with tap "T2" closed.

By carefully opening T2 the mercury in "A" is made to rise until the mark is reached. T2 is then closed. One or two ccs. of KOH solution is now drawn into "B." After driving out any gas bubble which may be present in "B," the gas is transferred to "B." Each reagent which is added to "B" should be gently agitated to assist absorption of the gas. When absorption is complete the gas is transferred to the burette for measurement. Tap T1 is reversed as soon as the reagent has reached the left hand side of T1, the reagent is driven out through "x" into a beaker which is placed to receive it, T1 then reversed again, and the gas followed up with mercury as far as "E."

Constant pressure method.—The gas sample is transferred to the bulb "B" and thence to the burette "A" in a similar manner as in the constant volume method, but where there is less tendency to leak it is more convenient to bring the mercury as far as the left of T1 only. (The graduations on the burette refer to the volumes contained between the marks and "E"; the volume of capilliary between "E" and the left of T1 is only about 0·02 mls.; thus, if the gas is measured in this way a correction of 0·02 mls. is to be added to the original volume, but for most purposes this is negligible).

Reagents are added to "B" and absorption made in a similar manner as in constant volume work. After the reagent has been driven out through "x," it is more convenient to bring the gas merely to the left hand side of T1. If this is done, the gas may be measured before expulsion of the reagent. The simplest and most accurate control in all these manipulations is obtained by the careful opening of T2.

General Maintenance of the Apparatus

The taps must be kept free from chemicals and well greased. A periodical cleaning (especially T1) should be made by wrapping a piece of filter paper round the tap and rotating it in its socket. A very thin smear of tap grease should be given, not enough to clog capillaries and interfere with combustion in the bulb "B"; at the end of each analysis, the burette and the vessel B should be washed out with dilute acid (approximately 2N sulphuric) by drawing a few mls. into B, transferring it to the burette, back into B and expelling it through "x." The apparatus must not be allowed to stand containing caustic alkali—the tap will either seize up or become permanently impaired. If the gases which are determined by combustion are other than hydrogen, the apparatus must be washed free from alkali before combustion. This is quite easily done by washing out B with acid and twice with water. About 1 cc. of water is then introduced into B and transferred to the burette; the water and gas are then transferred back to B, the gas transferred to the burette and the water expelled. No water should enter the burette containing gas from B until the latter has been washed free from acid, otherwise the traces of acid brought ove from B may react with traces of carbonate in the burette and generate carbon dioxide which will be recorded as methane after the combustion.

No reagents should be allowed to enter the burette as they remove water vapour from the gas, causing a false contraction in volume. Should this accidentally occur, the burette (after expulsion of the reagent) should be washed out with water, and the gas volume again measured.

Temperature changes during an analysis should be kept as low as possible; as little as 1° C. change causes a volume change of approximately 0·3%. A compensating device should be fitted if great fluctuations in temperature cannot be avoided.

Special Applications of the Ambler

One particular application is the analysis of flue gases. The carbon dioxide is determined by absorption and the total hydrogen and carbon monoxide then determined by combustion; there is usually enough air present in the sample to burn the small amounts of combustible gases, but if a case is met where a great excess of these combustible gases is present a further quantity of oxygen must be added for complete combustion. In the former case it is not necessary to remove the alkali solution since in the presence of alkalis both hydrogen and carbon monoxide give a contraction of 1½ volumes in combustion and for control work it is unnecessary to estimate them separately. The remaining oxygen can be determined by adding aqueous pyrogallol and absorbing. To obtain the total oxygen, a volume equal to one-third of the contraction on combustion is added.

Fractional Combustion

Providing the hydrogen plus carbon monoxide figure does not exceed 3%,

the fractional combustion of hydrogen and carbon monoxide can be carried out in the presence of methane. If necessary, air is added to the sample which is then introduced into B and the filament brought to a very dull red heat (just visible in daylight against a black background) and maintained at that temperature for one minute. Should the hydrogen plus carbon monoxide content be greater than about 2% the current must be turned on very slowly, otherwise the heat of combustion of these gases causes the methane to burn.

The whole of the hydrogen and the carbon monoxide are oxidised and the methane as yet unaffected may be separately determined by bringing the filament to a bright yellow heat for two minutes.

The Geology and Mineral Resources of Kenya

By W. Pulfrey, M.Sc., Ph.D., F.G.S., A.Inst.M.M.
(Senior Geologist, Mining and Geological Dept., Kenya)

THE seventh of the Imperial Institute's series of illustrated lectures on recent progress in geological investigations and mineral developments in the Colonies was devoted to the geology and mineral resources of Kenya and was delivered by Dr. W. Pulfrey. In his introductory remarks it was explained that although a number of geological investigations has been undertaken privately or by official geologists appointed for short periods or for specific investigations, it was not until 1932 that a permanent Geological Survey, incorporated in the Mines Department, was organised. It has functioned continually since that date, though usually with a diminished staff.

After giving an interesting description of the general physiographic features of the country—an area twice that of the British Isles—Dr. Pulfrey proceeded to discuss the nature and distribution of the principal groups of rocks. The oldest are those of the Basement System of Archaean age and consisting essentially of gneisses and schists which cover wide areas of the colony, particularly in the eastern half. Next in age are the pre-Cambrian (but post-Archaean) rocks occurring in the goldfields of western Kenya. Two systems have been differentiated, the Nyanzian, which is the older, and the Kavirondian System. The sediments and volcanics of the Bukoban System (Kisii Series), probably Palaeozoic, come next in age and are restricted to a small area in and east of southern Kavirondo where they give rise to the Kisii Highlands. Sediments of Palaeozoic and Mesozoic age occur along the coast, while in the north-eastern corner of the Colony occur other Mesozoic sediments, which cannot be correlated directly with those of the coastal belt. Tertiary deposits include small exposures of sediments in the coastal area and volcanic rocks and Miocene sediments of the Highlands, western Kenya and the Northern Frontier District. Pleistocene rocks are represented by raised coral reefs and some sandstones at the coast, sediments in the Rift Valley and some volcanic rocks of the Highlands. Recent deposits include soils and alluvial accumulations and saline deposits resulting from the desiccation of lakes such as at Lake Magadi.

Although numerous minerals have been discovered and exploited for export or for local use and have been a notable asset to the country, only two, gold and trona (as soda ash), have figured prominently as exports. Gold mining is restricted to areas in which the Nyanzian and Kavirondian Systems and the granites which invade them are represented, that is in the Nyanza Province and in a smaller area east of the Kisii Highlands. Although mining had been carried out in a small way for several years in a belt near the Tanganyika boundary, it was not until 1931 that the Kakamega goldfield was discovered. The discovery led to a more intensive investigation and exploitation of the deposits of the whole of Kavirondo. Gold mining was soon on a sound economic footing with a steadily increasing output until the difficulties of the war years caused a gradual decline. A maximum production of a total of 104,976 unrefined ounces, valued at £607,753, was achieved in 1939 falling in 1945 owing to war conditions to 38,517 refined ounces, valued at £333,259. The total value of the gold produced up to 1945 was £4,952,098.

The value of other minerals produced (in 1945, totalling £73,734) is small compared with that of the two chief mineral exports. They include asbestos (anthophyllite and crocidolite), barytes, bentonitic clays, brick earths and clays, beryl, building stones, diatomite, felspar, garnet, graphite, gypsum, iron ores, kaolin, Kisii soapstone, kyanite, galena, limestone, magnesite, manganese ores, mica, pumice, pyrite, pigments, quartz, sapphire, talc and vermiculite, which have been or are being worked on a small scale principally for local use. Other minerals which are known include: Mispickel, copper ores, dolomite, mineral aggregates with pozzuolanic properties, ilmenite, alum, amblygonite, apatite, cassiterite, cinnabar, columbite, gemstones, molybdenite, monazite, nepheline, scheelite, native sulphur and zircon, but it is doubtful if many of these will prove of economic importance.

Although the mineral production of Kenya has not been of outstanding importance in the past, it must be remembered that much of the Colony has not been geologically surveyed and what prospecting has been undertaken has often been inadequate owing to insufficient finance or lack of experience. With the expansion of geological services it is not unlikely that mineral discoveries will become more frequent, and it is hoped that a stable and valuable mineral industry will arise.

In conclusion, Dr. Pulfrey outlined plans for the future. An immense amount of work both in the field and in the laboratory, is necessary, and in order to be of maximum value in post-war economy, must be carried out reasonably quickly and the results published. Since 1945, progress has been made in the construction and equipment of a mineral and ore-testing laboratory. A scheme for detailed and reconnaissance surveys of the greater part of the southern half of the colony has been initiated and, as staff becomes available, the mapping of potentially mineralised areas as well as of areas in which economic minerals are already known to occur, will be undertaken.

MICROANALYSIS
CHEMICAL AND PHYSICAL METHODS
APPARATUS METALLURGICAL APPLICATIONS TECHNIQUE

IN view of your recent remarks in these columns on comparative organisation of chemists in this country and in the United States of America, we have been reading with some interest the Report on the Organization of the American Chemical Society which was published a few months ago, and which has just reached us. This fifty-seven page Report contains much of note. It is based on a survey of membership opinion prepared by the Opinion Research Corporation, and contains a detailed analysis of replies to thirty questions, posed to a cross-section of the members. The first point of interest is that in the A.C.S., more closely integrated than anything in this country, a desire for improvement in organisation is expressed. It is doubtful if we have, at the moment, in this country, any machinery for assessing a similar requirement here, or for putting it into effect if expressed. The second point which interests us, as agreeing with views frequently expressed by us, is the emphasis placed on regional meetings and the tendency to favour these in preference to national meetings. Finally, of the members questioned fifty-seven per cent., or a majority, read or refer regularly to the Analytical Edition of Industrial and Engineering Chemistry. This figure of course, emphasises the excellence of the journal in question. But it is also—and this is of especial importance to us—a silent commentary on the importance of analytical chemistry in the scheme of things where any overall view of the profession is being considered.

Amperometric Titration

Part III.—Use of a Rotating Micro-Electrode

By J. T. Stock

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The general principles of amperometric titration, a technique closely related to polarography, have been discussed in previous articles. Although most of the amperometric determinations so far developed employ the dropping mercury electrode, the utility of the rotating platinum micro-electrode has been clearly demonstrated. The rotating micro-electrode is particularly useful when dissolved oxygen does not interfere, so that the titration may be carried out in an open vessel.

PARTS I and II of this series[1, 2], in which the general principles and instrumental aspects of amperometric titration were discussed, dealt almost exclusively with titrations which were performed with the aid of the dropping mercury electrode. It is now proposed to discuss the use for this purpose of the rotating platinum electrode and to consider some of its applications.

Though current-voltage phenomena with rotating electrodes have been studied by numerous workers[3], it was not until 1941 that a more exact investigation was carried out by Laitinen and Kolthoff[4]. These workers showed such electrodes could also be used in certain amperometric titrations. Apart from a single publication[5], no further news of the use of the rotating electrode has been received until the present year. Recently, however, details of several amperometric titrations which may be simply and elegantly performed by the aid of this electrode have been published: others will doubtless follow.

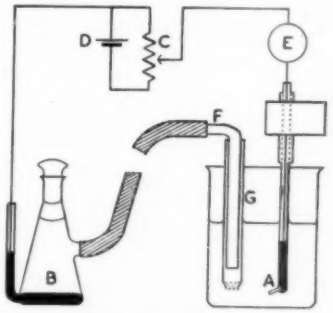
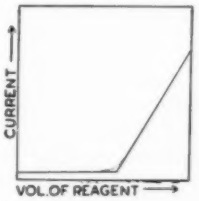


Fig. 1.—Apparatus for amperometric titration with a rotating platinum micro-electrode.

Fig. 2.—One form of titration curve.



1 J. T. Stock, *Metallurgia*, 1944, **30**, 221.
2 J. T. Stock and M. A. Fill, *Ibid*, p. 277.
3. For a review, see I. M. Kolthoff and J. J. Lingane, *Polarography*, Interscience Publishers, Inc., New York, 1941, p. 437.
4 H. A. Laitinen and I. M. Kolthoff, *J. Phys. Chem.*, 1941, **45**, 1079.
5 P. A. Giguère and L. Lauzier, *Canad. J. Res.*, 1945, **23B**, 223.

The apparatus for the performance of an amperometric titration is depicted schematically in Fig. 1. The rotating platinum micro-electrode A and the salt bridge connecting to the reference electrode B are arranged to dip into the solution to be titrated. Potentiometer C, energised by battery D, enables the potential of the micro-electrode to be adjusted so that the *diffusion current*[1] of the substance to be determined, or of the reagent, will be obtained. The current flowing is measured by galvanometer E. Since the pulsations in current which occur when using the dropping mercury electrode are not experienced with the rotating electrode, a sensitive micro-ammeter may be used in place of a galvanometer.

Having set the rotating electrode in motion at about 600 r.p.m., the current reading is noted and portions of the reagent are added from the burette, reading the current after each addition. For example, in the titration of bromide with silver nitrate solution the current is initially very small, since the bromide ion does not form a polarographic wave under the experimental conditions. As the titration proceeds, little alteration in the current strength is observed until the end point is reached. Further addition of the reagent introduces into the solution an appreciable concentration of silver ions. These are wave-forming, so that the current now increases as the titration is continued. On plotting the current readings against the corresponding volumes of reagent added, a titration curve of the type shown diagramatically in Fig. 2, consisting of two straight lines intersecting at the end point, is obtained. (Part I should be consulted for other forms of titration curve, and for deviations of the curve from the ideal shape [1]).

The rotating electrode consists merely of a piece of platinum wire about 6–8 mm. long and 0·5 mm. in diameter. This is sealed through the closed lower end of a length of 6 mm. glass tubing, which forms the stem, as shown at (*a*) in Fig. 3. The region of the glass-to-platinum seal should be carefully examined for minute cracks; if any are found, a new electrode should be prepared. For titrations in which interference with the current readings is occasioned by the presence of much suspended material in the solution, a modified design of electrode may be used. This is shown in elevation and top view at (*b*) and (*c*), respectively, in Fig. 3, and its use eliminates such interference[6]. Laitinen and his co-workers[7, 8] use a No. 18 platinum-wire electrode sealed into glass tubing which is then bent at right angles about 2 cm. from the end, as shown at (*d*) in Fig. 3.

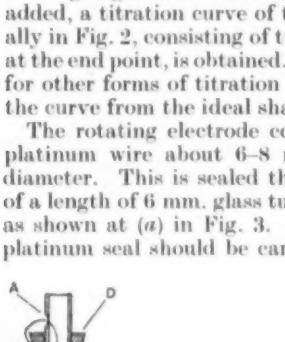


Fig. 5.—Sleeve and well method of making electrical connection.

The method of making electrical connection between the platinum micro-electrode and the electrical apparatus is most important. Sliding or rubbing contacts should be avoided[7]. One method is to use a stirrer with a hollow shaft. A piece of copper wire, the remote end of which is connected to the remainder of the apparatus, is then thrust down the bore of the shaft, to dip into a bead of mercury in contact with the inner end of the platinum wire, as shown at (*a*) in Fig. 4. To employ an ordinary cone drive motor, its shaft may be replaced by a short length of brass tubing within which the stem of the rotating electrode is secured[9]. Contact is then made as before.

When this type of stirrer is not available, the method shown at (*b*) may be employed. A bead of mercury or a small piece of Wood's metal enables the inner end of the platinum wire to make good connection with a piece of copper wire, the upper end of which is connected to the stirrer shaft. The upper end of the latter is drilled to form a small cup which contains a drop of mercury. A copper wire dipping into the latter then allows satisfactory connection to be made to the electrical apparatus[4].

A third method of making connection is shown in Fig. 5[10]. A small hole A is blown in the upper part of the electrode stem B and allows emergence of the copper wire which makes contact with the inner end of the platinum wire by the agency of a bead of mercury. The

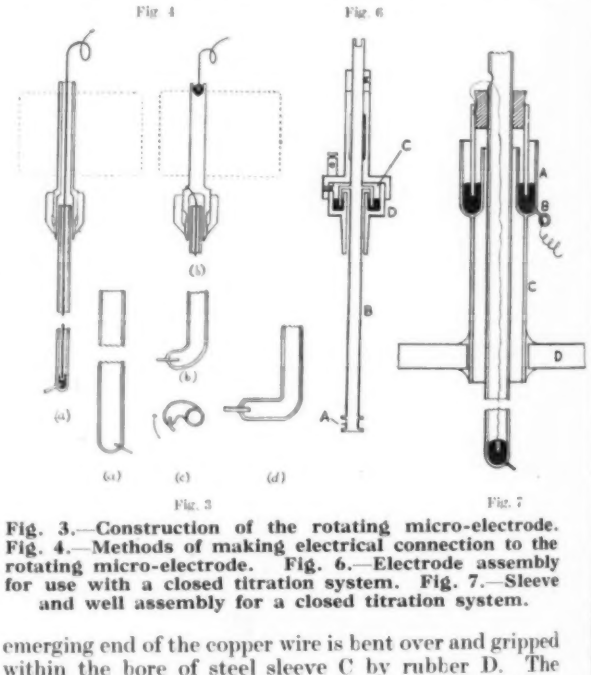


Fig. 3.—Construction of the rotating micro-electrode. Fig. 4.—Methods of making electrical connection to the rotating micro-electrode. Fig. 6.—Electrode assembly for use with a closed titration system. Fig. 7.—Sleeve and well assembly for a closed titration system.

emerging end of the copper wire is bent over and gripped within the bore of steel sleeve C by rubber D. The latter is cut from pressure tubing and holds the sleeve in position upon the electrode stem. Well E is also of steel and contains mercury, into which dips the lower end of the sleeve. A shank F is welded to the well and enables the latter to be supported on a retort stand by means of a boss-head, a sleeve G of rubber tubing serving to insulate the shank from the boss-head. Connection is made by attaching a wire to terminal H. To avoid rusting, all portions of the steelwork not actually required for contact purposes are coated with cellulose enamel.

The electrode assemblies described above are satisfactory for titrations in which dissolved oxygen does not interfere and in other cases when an open titration

6 I. M. Kolthoff and W. E. Harris, *Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 161.
7 H. A. Laitinen, W. P. Jennings and T. D. Parks, *Ibid*, p. 355.
8 *Ibid*, p. 358.
9 Weiblen, D. G., quoted by I. M. Kolthoff and W. E. Harris, *Ibid*, p. 161.
10 J. T. Stock, *Unpublished experiments.*

vessel may be used. Modification is necessary if a closed titration system is required. For such a system, the all-metal electrode assembly shown in Fig. 6 is suitable[4]. The platinum micro-electrode A is attached to an iron shaft B; the flanges either side of the electrode protect it from damage when the shaft is inserted into a rubber stopper. An inverted cup C is formed upon the shaft and dips into the mercury in well D. This allows satisfactory electrical contact to be made and also prevents ingress of air into the titration vessel. A geared synchronous motor operating through a flexible shaft which engages in the slot at the top of shaft B enables the latter to be rotated at constant speed and with the minimum of vibration. To protect the iron shaft from corrosion and to insulate it, several layers of ceresin wax are applied, the wax being carefully removed from the platinum electrode.

Another method suitable for use with a closed titration system is shown in Fig. 7[10]. The rotating electrode, steel sleeve and rubber stopper are identical with those in Fig. 5. A glass mercury well A is employed, connection to the electrical apparatus being made by attaching a wire to platinum loop B, the shank of which is sealed through the wall of the well. The lower end of extension tube C is cemented into a hole in the bakelite cover D of the titration cell. Holes in the cover are also provided for the introduction of the jet of a micro-burette, tubes for entry and exit of nitrogen or hydrogen, etc.

Though a salt bridge of normal form may be used to connect the reference electrode and the solution to be titrated, the flexible form shown in Fig. 1 is particularly convenient.[11] It consists of up to 2 ft. of 6mm. bore soft rubber tubing which is filled with saturated potassium chloride solution. The end of the tubing remote from the reference electrode vessel[6] is connected to a short length of glass tubing F filled with a 3% agar—30% potassium chloride gel. (Care must be taken to eliminate air bubbles, which would increase the electrical resistance). Tube G, the lower end of which is closed by an agar—or a fine sintered glass plug, is interposed between the end of the salt bridge and the solution to be titrated. The contents of this tube may be readily rinsed out when required, thus avoiding contamination of the test solution.

Use of the rotating electrode is obviously simpler when the titration may be carried out in an open vessel—e.g., if interference by dissolved oxygen does not occur. The latter form of interference does not occur if the rotating electrode is impressed with a positive or *low* negative potential. Such potentials may, of course, be applied by potentiometer C (Fig. 1); low potentials may also be obtained by suitably choosing the reference electrode. Potentiometer C and battery D are then unnecessary, the galvanometer merely being connected between the rotating electrode and the reference electrode.

An example of this technique occurs in the titration of *n*-dodecyl and other mercaptans with silver nitrate solution[6]. This technique allows as little as 0·2 mg. of mercaptan sulphur in 100 ml. of ethanol to be determined with an accuracy of from 1–2%; with amounts greater than 2 mg., the accuracy is at least 0·3%. The titration is carried out in the presence of ammonia, so that large amounts of chloride and small amounts of bromide may be present without interference. A titration curve of the type shown in Fig. 2 is obtained. The filling of the reference electrode consists of a layer of mercury in contact with saturated potassium chloride solution containing potassium iodide and mercuric iodide. Use of this system, which has a potential of –0·23 volt with reference to the saturated calomel electrode, enables the battery and galvanometer to be dispensed with.

Since mercaptans may be extracted by ether or other suitable solvent, they may be readily separated from interfering ions. This method of separation is useful when cyanide, iodide or sulphide are present, since these substances interfere with the titration.

Alkyl disulphides[12] may be reduced by amalgamated zinc to mercaptans, which may then be titrated in ammoniacal solution as described above. The reduction is carried out at 50° C. in 90% alcohol which is 0·3 *N* in sulphuric acid (or in acetic acid when the amount of disulphide is small) by passing through a Jones reductor arranged for upward flow. Alternatively, the solution and amalgam may be placed in a screw-cap bottle which is rotated for 20 minutes in a bath maintained at 50° C. Incompleteness of the reduction limits the accuracy of the method; the results are about 5% low, but reproducible to 2%. Less than 1 mg. of disulphide may be determined by this method. Mixtures of disulphide and mercaptan may also be analysed by titrating portions of the solution before and after reduction, the difference representing the amount of disulphide present. When the amount of mercaptan greatly exceeds that of disulphide, the difference method cannot be used. Instead, the solution is very carefully titrated amperometrically until a very slight excess of silver is present. The precipitated silver mercaptide is then filtered off and the disulphide in the filtrate is reduced with zinc amalgam and titrated as before.

The amperometric titration of chloride with silver nitrate solution was studied by Laitinen and Kolthoff[4], who obtained excellent results if gelatin was added to the solution. This substance prevents depolarisation of the electrode by colloidally-dispersed particles of silver chloride. The work was later extended by Laitinen, Jennings and Parks[7], who showed that the titration in aqueous solution is satisfactory down to 0·002 *N* with respect to chloride. Addition of acetone is recommended when the concentration of chloride is low; in a medium which contains 75% of acetone the error in titrating 0·0001 *N* chloride is 3%. Bromide and iodide may also be titrated, and mixtures of halides may be analysed[8]. To do this, advantage is taken of the following facts: (i) Iodide may be successfully titrated in a dilute ammoniacal solution, chloride and bromide not interfering. (ii) In the absence of gelatin, silver chloride depolarises the platinum micro-electrode, whereas silver bromide and iodide do not. The solution containing the three halogens is therefore initially rendered ammoniacal and is titrated to a first end point which corresponds to the amount of iodide present. After acidification, the titration is continued to the bromide end point. Gelatin is then added; a final titration gives the amount of chloride present.

The rotating electrode may also be used for the amperometric titration of arsenious oxide with potassium bromate solution[4]. A titration curve of the type shown

11 N. Hume and W. E. Harris, *Ind. Eng. Chem., Anal. Ed.*, 1943, **15**, 465.

12 I. M. Kolthoff, D. R. May, P. Morgan, H. A. Laitinen, and A. S. O'Brien, *Ibid*, 1946, **18**, 442.

in Fig. 2 is obtained. The accuracy is high, being 0·1% and 0·3% for solutions which are respectively 0·001 *N* and 0·0001 *N* with respect to arsenic trioxide.

An interesting titration is that of acidified chromate solution with ferrous iron solution[13]. The rotating electrode is used as the *anode*, being maintained at a potential of + 1·0 volt with reference to the saturated calomel electrode. Since the chromate ion forms no polarographic wave under these conditions, the initially small current shows little or no increase until the end point has been passed. Further addition of reagent then introduces ferrous ions, which, owing to oxidation to the anodic state, give rise to a polarographic wave. Continuance of the titration thus causes a rapid increase in the flow of current, as in previous cases. The method is rapid and is also accurate and precise even when the concentration of chromate is only about 0·0001 *N* or 0·0002 *N*. The reverse titration of traces of ferrous iron with dichromate is also possible: the current then decreases during the process, becoming zero at the end point.

13 I. M. Kolthoff and D. R. May, *ibid*, p. 208.

Miscellaneous Microchemical Devices

XI.—Ground Joints for Small-Bore Tubing

By J. T. Stock and M. A. Fill

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IN a micro-scale investigation of the oxygen uptake of edible oils (which is still proceeding), it became necessary to devise a means whereby the various portions of the apparatus could be readily assembled without introducing much free space into the system. Simple ball-type joints, the halves of which are held in contact by a tensioning device cut from rubber tubing, were found to be satisfactory, and may be used to connect tubes of from 0·2–3 mm. bore without the increase in dead space usually associated with cone-type joints.

As shown at (*a*) in Fig. 1, the halves of the joint are formed upon the ends of the tubes to be connected. The male half is formed merely by fusing the end of the tube in a blowpipe flame, a stream of air blown through the bore preventing the latter from becoming constricted. To form the female half, a copper tool made from 18-gauge copper sheet, as shown at (*d*), may be used. Alternatively, the end of the tube may first be closed by fusing, when a small, thick-walled bulb is blown and caused to burst. After fire-polishing, a smooth cup-shaped cavity is obtained. The second method of construction is particularly suitable for jointing tubing of bore less than 1·5 mm.

After forming, the halves of the joint are ground together with fine emery powder and water. A convenient method is to hold one-half of the joint by its stem in the chuck of an electric stirrer which is running at low speed, and to apply by hand the second half. The latter should be given a rocking motion to produce a spherical seating. Having removed the abrasive, the halves are examined and may be lapped together with rouge. The tensioning device consists of a short length of soft rubber tubing which firmly grips the walls of the tubes as shown at (*b*). Two diametrically-opposite holes, cut with a sharp cork-borer, relieve the inward pressure in the immediate vicinity of the joint and allow a reasonable degree of flexibility without causing the halves to pull apart. A well-ground joint, the halves of which are lightly greased before assembly, will remain sound not only under vacuum but also under a pressure of from one-half to one atmosphere.

When desirable, the design may be modified to incorporate a mercury seal. The simplest manner of doing this is to increase the size of the female half, as shown at (*c*), when a few beads of mercury may be introduced after the joint has been assembled. Alternatively, mercury cups may be sealed on to a joint of normal construction, as shown at (*e*). The rubber tubing providing the tension is then stretched between the parallel portions of the mercury cups, as shown in broken lines. Though less simple, the second method of construction is preferable, the large diameter of the rubber tubing bestowing a self-centering effect upon the joint.

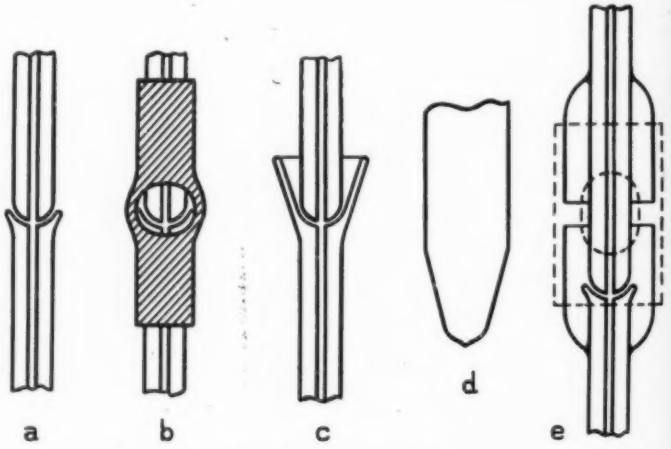

Fig. 1.—Miniature ball-type joints.

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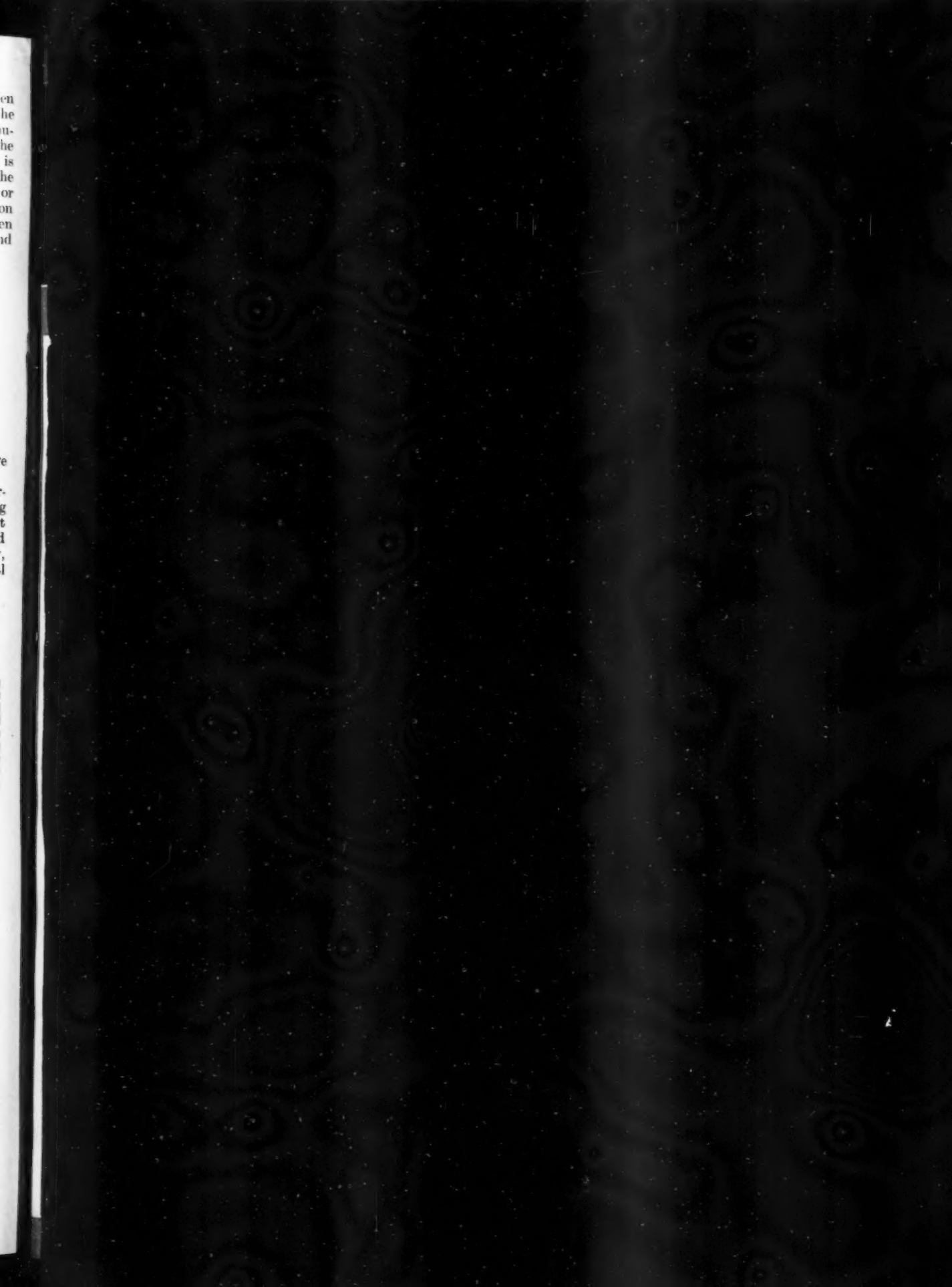
This Company states that the illustration of a die, which appeared in the article on Page 301 in the last issue of "Metallurgia," was made from a photograph supplied by Messrs. Almin Ltd. the well-known Light Metal Industries Group.